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Ultra deep SU-8 manufacturing and characterization for MEMS applications

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ULTRA DEEP SU-8 MANUFACTURING AND CHARACTERIZATION FOR MEMS APPLICATIONS

A Thesis

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Abstract

The Micro Systems Engineering Team (μSET) at Louisiana State University (LSU) utilizes microfabrication for a number of heat and mass transfer devices. These include cross flow heat exchangers, mechanical seals with integrated micro heat exchangers, catalytic converters, and micro reactors. In all of these applications, micro honeycomb arrays provide increased surface area per unit volume which significantly enhances heat and mass transfer. In the past, it was only possible to fabricate SU-8 structures approximately 1.5 mm tall. Furthermore, qualitatively, it is much more difficult to fabricate close packed feature arrays than sparse arrays. For many of the previously mentioned applications, it is important to both increase the height of the features and to produce considerably more closely packed features.

The goal of this research is to develop a greatly enhanced capability to lithographically define SU-8 features with heights that are on the order of 2-3 mm, with characteristic widths that are on the order of a few hundred micrometers, and, equally important, close packed. The major discovery that was ascertained in an attempt to achieve this goal was the diffusion of acid into unexposed regions prior to and during post bake is <u>THE</u> important physical parameter that governs all SU-8 processing steps. From this central idea, all SU-8 processing steps were altered to limit diffusion. The main process modification that allowed for this accomplishment was the new casting procedure that permitted for low uniform solvent content. The resulting new processing procedure led to SU-8 samples with heights between 2-4.5 mm and with a high density of SU-8 structures.

1. Introduction

Ongoing research efforts both at LSU and Mezzo International Technologies focus on fabricating components such as mechanical seals, heat exchangers, regenerators for cryo coolers, and high cell density catalyst cores that incorporate micro scale features to achieve extremely high heat/mass transfer rates per unit volume (or unit weight) compared to conventional scale counterparts. In all these applications, a mold tool with high aspect ratio micro features (HARMs) is used to mass produce parts. For the applications listed above, improved performance is invariably associated with increasing the absolute height of the micro features, (values of 2-5 mm are desired) while maintaining high aspect ratios (height/width ratios between 10 and 30). Finally, if possible, the features should be tapered to facilitate demolding [15].

The LIGA process, via deep x-ray lithography using SU-8, potentially provides a cost effective means to electroform molds needed to mass produce parts with micro features having heights between 2-5 mm and aspect ratios of 10-30. To electroform such molds, ultra tall, often densely packed SU-8 features must first be lithographically defined. SU-8 has absorption characteristics very similar to the most common resist used in x-ray lithography, PMMA, but it much more sensitive. For the case of the available radiation spectra available at Center for Advanced Microstructures and Devices (CAMD) in Baton Rouge, the time required to expose an SU-8 sample of given thickness is over 100-fold less than that required for PMMA [2]. For the last few years, SU-8 features with heights on the order of 1-1.5 mm have been routinely lithographically defined [16].

Unfortunately, lithographically defining SU-8 features with aggressive combinations of heights, aspect ratio, and feature density has been problematic. The absorption of photons within the SU-8 produces a photoacid generator (PAG). A post bake at an elevated temperature (95 °C is an often-referenced value) provides sufficient energy for the PAG to initiate cross linking that makes the SU-8 insoluble in developer. The main premise of this thesis is that the diffusion of acid into unexposed regions prior to and during post bake is the most important physical phenomena that controls whether or not SU-8 features can be lithographically defined. If sufficient acid diffuses into the unexposed regions, undesirable cross linking will occur in nominally unexposed regions. By controlling a number of factors that affect diffusion, very tall, dense patterns of SU-8 features can be lithographically defined.

1.1 X-Ray Lithography

The Microsystems Engineering Team at Louisiana State University (LSU) has for the last seven years used a three-step process known as LIGA to successfully produce microstructures useful in many fields (Figure 1). The first step, X-ray lithography (LI), is used to pattern a photo resist, usually (poly)methylmethacrylate (PMMA) or SU-8, by the use of collimated radiation. This radiation changes the molecular pattern in the photo resist, either by cross linking the molecular bonds in negative resists (SU-8) or break the bonds and reducing the molecular weight of a positive resist (PMMA). An x-ray mask consisting of a pattern of gold absorber features supported by a graphite membrane that is transparent to x-rays is used to lithographically filter incident radiation onto a sheet of x-ray sensitive resist that is mounted on a substrate (in this study the substrate is a stainless steel plate of thickness 0.25 inch). The exposed photo resist is then immersed into a developing solution, and in the case of a negative resist, the unexposed sections of resist are removed from the substrate leaving the desired structures behind. For the positive resist the opposite is true. The exposed resist is removed from the substrate leaving the untouched resist as the desired microstructures.

The second step in the LIGA process is galvanoformung or electroplating (G). The electrically conductive substrate with the remaining resist pattern is then submerged into an electroplating bath. Metal is deposited between the voids in the resist structures until the voids are completely filled. Once the plating fills the voids, the deposition is no longer constrained by the resist features and if the electroplating process is continued, the features will merge and form a continuous plate that is parallel to the original substrate from which the electroplating process originated. After the over plated layer becomes sufficiently thick, the electroformed part is debonded from the original substrate (the bond between substrate and deposited metal is weak). The resist is removed from the electroformed part and the result is a metal structure with the negative desired feature pattern built in.

The final step in the LIGA process is abformung (A) or molding. The electroplated metal structure is then used as a mold insert in one of many processes, including embossing and injection molding. In the case of injection molding, the part is mounted into the machine and plastic is injected into the feature pattern and the desired plastic microstructures are produced.

1.2 SU-8

The three most important parameters that define resist performance are sensitivity, contrast, and absorptivity. Sensitivity defines the exposure dosage that must be absorbed to effect the necessary change in solubility during the subsequent

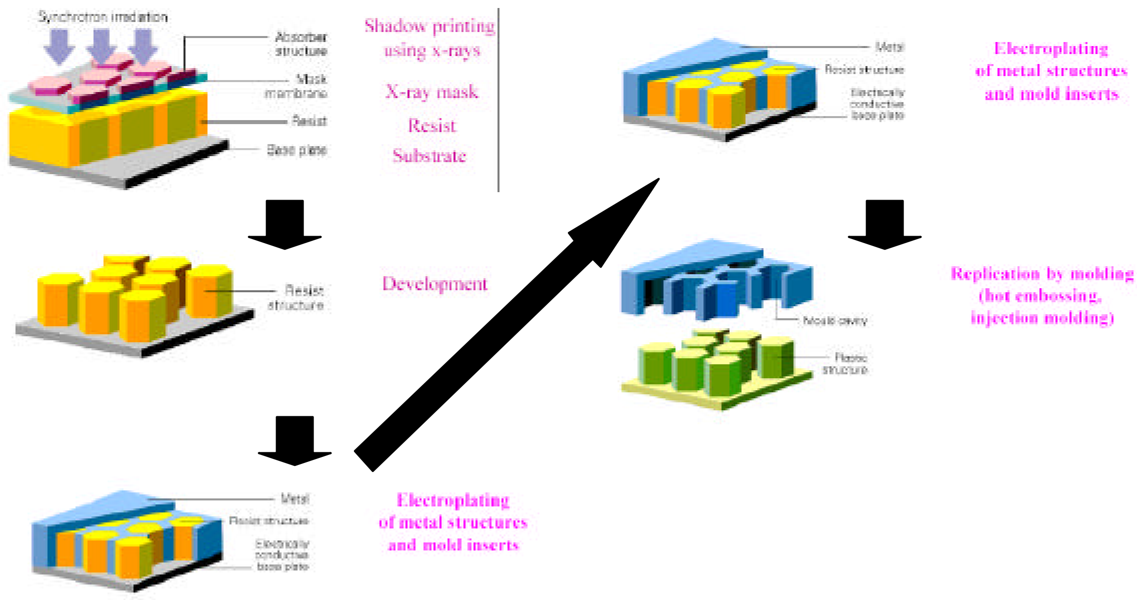


Figure 1: LIGA process: Lithography, developing, over plating, and molding.

development process. All factors being equal, the required exposure time is inversely proportional to the sensitivity. Therefore, a high sensitivity is desired. Contrast is the rate of change of development with respect to molecular weight (for the case of a positive resist) or degree of cross linking (in the case of a negative resist). Absorptivity is defined by the rate of the exponential decay of radiation passing through the resist. Too high an absorptivity limits the thickness of the resist layer that can be exposed, while too low an absorptivity produces excessive exposure times. Table 1 below provides a comparison of the exposure times of SU-8 and PMMA using one of the bending magnet beamlines at the CAMD, Louisiana, U.S.A [2]. The exposure times were calculated for SU-8 and PMMA assuming bottom doses, respectively, of 15 J/cm^3 and 3000 J/cm^3. For the PMMA cases, sufficient aluminum filters are used to maintain the top-to-bottom dose ratio to less than or equal to ten.

Table 1: Comparison of the typical exposure times of PMMA and SU-8 resists of various thicknesses using an X-ray mask with a 320 mm graphite membrane mask. Ring energy = 1.3 GeV; bending magnet radius = 2.928 m; average ring current = 100 mA; scan length = 4 cm; distance from the source = 10 m [15].

Resist thickness	SU-8 exposure time (minutes)	PMMA exposure time (minutes)	Aluminum filter thickness for PMMA	PMMA top-to-bottom dose ratio
2000 µm	15.3	5452	50 µm	8.1
1500 µm	9.5	2802	30 µm	7.6
1000 µm	5.3	1408	20 µm	5.5
500 µm	2.4	400	none	4.1

The other advantage of SU-8 is associated with its use in a *proven* process to produce mold tools having microfeatures with *tapered* sidewalls [15]. The economic viability of the LIGA process is based upon mass production via injection molding or embossing parts using a mold tool. Demolding is greatly facilitated if the sidewalls are tapered. Virtually all-conventional scale mold tools incorporate tapered sidewalls. SU-8 high aspect ratio features with tapered sidewalls have been lithographically defined using a multiple exposure tilt-and-rotate process [15]. A subsequent electroforming step results in a mold tool with tapered sidewalls. A similar process using PMMA has not yet been demonstrated. Therefore, SU-8 has two important advantages with respect to PMMA: higher sensitivity and the proven ability to fabricate mold inserts with tapered sidewalls that are absolutely crucial for molding features with heights above 1-2 mm [15].

There are some advantages of PMMA in comparison to SU-8. PMMA features have higher dimensional tolerances then that which can be defined by SU-8. Since the typical structure size will be 100's of micrometers across, the tolerance capability is a second order parameter.

In the implementation of the LIGA process the resist must be removed after electroforming. PMMA may be easily removed by the use of acetone, while SU-8 must be burnt out. The ease at which PMMA may be removed is not a significant advantage due to the fact that the burn out procedure does not cause detrimental effects to the mold insert.

SU-8, which is sold by Shell Chemical and developed by IBM, is the main constituent of negative tone resists [9]. SU-8 is the combination of a resin, solvent, and photoacid generator (PAG). The resin is an epoxy made up of a bisphenol A novolac glycidyl ether. On average, there are 8 epoxy groups in a typical molecule, hence the “8” in SU-8. This is a typical molecule because in reality molecules exist in a number of different size and shapes. The organic solvent used is gamma-butyrolacton (GBL), which varies in concentration depending on desired viscosity. Triarylsulfon salt is the chemical that comprises the PAG.

The PAG releases acid after it absorbs a photon. Thus only regions that have been exposed with a light source have an acid present. A heating process is then required to give the reaction the energy necessary for cross linking to occur. The combination of heating and the presence of acid allow the SU-8 to cross link. The cross linked SU-8 is insoluble in developer while the rest is not. The change in size and density is problematic when exact images must be formed and when there is a need to remove the cross-linked resist. However, these problems can be overcome if they are taken into account before hand. Exposed SU-8 may be removed by the use of a burnout procedure and the shrinking of SU-8 can be taken into account by properly over sizing the x-ray mask to take account for shrinking.

1.3 Goal

The goal of this research is to develop a greatly enhanced capability to lithographically define SU-8 features with heights that are on the order of 2-3 mm, with characteristic widths that are on the order of a few hundred micrometers, and, equally important, close packed. These feature patterns are useful for a number of heat/mass transfer applications that are being developed in the Micro Systems Engineering Team Laboratory (µSET). These include cross flow heat exchangers, mechanical seals with integrated micro heat exchangers, catalytic converters, and micro reactors. In all of these applications, micro honeycomb arrays provide increased surface area/unit volume that enhances heat/mass transfer. In the past, it was only possible to fabricate SU-8 structures approximately 1.5 mm tall. Furthermore, qualitatively, it is much more difficult to fabricate close packed feature arrays than sparse arrays. For many of the previously mentioned applications, it is important to both increase the height of the features and to produce much more closely packed features. To achieve the desired, much more aggressive combination of height and feature density, a more thorough understanding is required with respect to the parameters that govern the processing of SU-8 as a deep x-ray resist.

1.4 SU-8 Processing

The four major steps in SU-8 micro manufacturing are listed below.

1. Pre-bake: After the SU-8 is applied to the surface of a substrate (either cast as described in this thesis, or spin coated), the SU-8 is placed in an oven or on a hot plate and baked to remove excess solvent.

2. Exposure: SU-8 is exposed to x-ray radiation in order to pattern the SU-8. As stated before, the PAG creates an acid in the exposed areas.

3. Post-baking: The SU-8 is placed in an oven (or on a hot plate) and as a result of being at a higher temperature for a given period of time, the SU-8 cross links where acid is present.

4. Development: An organic solvent is used to dissolve the unexposed area, leaving the patterned SU-8.

There are two ways that SU-8 may be coated onto a surface, spin-coating and casting. Spin-coating is the most widely used method. It is used in order to get flat thin layers of SU-8. For a single spin the highest SU-8 that has been achieved is ~1 mm [6]. Thicker layers are achieved by a multi-spin and baking process. After the first layer is spun, it is then baked to prevent reflow. On top of this layer, the process can be repeated a number of times to achieve higher thickness.

The alternate method to create SU-8 on a substrate is casting. A retention wall is used to hold the SU-8 on the specific area. After the SU-8 has been poured into this restricted area, the sample is baked. Samples of any height can be created using this process. The highest cast samples that have been previously created are 3.7 mm [6].

No matter which method of putting the SU-8 on the substrate is used, it must be baked to remove excess solvent. Through studies, it has been found that the optimal amount of remaining solvent is approximately 7 % [5]. The small amounts of solvent reduce stress cracks that can occur [14]. The two variables that control solvent content are time and temperature of the pre-bake. MicroChem recommends the pre-bake start at 65 °C and then ramp the temperature of 95 °C. The times held at each position are dependent on the thickness [11]. Temperatures can range from 60°C to 140 °C [12,14]. Times and temperatures vary widely from user to user. The times are even more varied then the temperatures. Some procedures use ramping, while varying the times by multiple hours.

The pre-baked sample is typically exposed with an ultraviolet (UV) or x-ray source. UV is a much cheaper exposure source, but x-rays give better results especially for thick resist layers. X-rays have a shorter wavelength then UV. This gives more precise feature tolerances and can create taller features. X-ray exposures have been reported with aspect ratios (height to width ratio) as high as 360 [1, 6]. UV can generate results in reproducible aspect ratios of 25:1 [1].

The reported optimum x-ray exposure doses range anywhere from 10-52 J/cm^3 [1]. This is the dose that the bottom of the SU-8 experiences. The dose within the sample is not uniform because the top of the sample absorbs energy. This makes the top of the sample have a higher dose then the bottom. The reported optimal doses are defined for specific geometries and thicknesses.

After the exposure is complete, the sample is heated to allow the acids to cause the cross-linking of the SU-8. The temperatures and times used in the post-exposure bake are just as varied as the procedures for the pre bake. Microchem suggests a ramping bake from 65°C to 95 °C. Holding times at these temperatures depend on the individual specimen [11].

Finally, the sample is placed in an organic solvent that dissolves the unexposed SU-8. Development is similar to the other steps in that there are many methods that have been used. The sample can be immersed with no agitation, stir bar agitation, ultrasonic

[6], or megasonic. With any of these techniques, heating the solution and refreshing the solution are options [1].

1.5 Governing Ideas

The previous references give a variety of SU-8 processing recipes, each of which only applies to a narrow range of SU-8 thickness/feature geometry combinations. The main premise of this thesis, to place these disparate results in context, is that the diffusion of acid into unexposed regions prior to and during post bake is THE important physical parameter that governs all SU-8 processing steps. Large amounts of acid diffusing into the unexposed regions will cross link this SU-8 and prevent it from being developed. Diffusion rates are a function of all of the processing parameters. In order to demonstrate that diffusion of acid in SU-8 can occur over time/length scales associated with a typical exposure, a one-dimensional model was constructed. The physics that governs the rate of acid migration is quite complex. The model assumes that within the exposed regions, the concentration of acid varies only with depth and that the concentration of acid within the unexposed regions is zero (very reasonable assumptions). To determine the concentration of acid within the unexposed region as a function of space and time, the model assumes that:

1. The concentration at the unexposed/exposed boundary at any elevation is constant (does not change during the post bake).
2. The diffusion of the acid into the unexposed region is governed by Fick's Law where the diffusivity of the SU-8 in the unexposed region does not vary with time. With these assumptions, the concentration within a channel of width 2W is given as a function of x and t by the Equation 1 below:

$$C(x,t) = (C_i - C_\infty)\sum_{n=0}^{\infty}\frac{2}{p\,(n+\frac{1}{2})}\sin\left(\frac{p}{L}\left(n+\frac{1}{2}\right)x\right)e^{-D(\frac{p}{L}(n+\frac{1}{2}))^2 t} + C_\infty$$

Equation 1

where:

$C(x,t)$ = the concentration as a function of space and time

C_i = the initial concentration in the unexposed gap

C_∞ = the concentration at the unexposed gap and exposure boundary

L = half of the unexposed gap's length

D = diffusivity of the SU-8

x = distance into the gap of the unexposed area

t = duration of diffusion

In reality, the modeling assumptions are incorrect. The model above is based on the implicit assumption that the acid diffuses through the unexposed SU-8, *then* reacts such that the degree of cross linking is proportional to the acid concentration. However, one would expect, that the diffusion and cross linking processes occur simultaneously. The cross linking process could affect both the concentration profile (by absorbing the acid) and the local diffusivity of the material. Also, the concentration of acid at the unexposed/exposed interface might be expected to decrease with time. Whatever diffusion range values that are obtained will be conservative. Figure 2 plots the spatial concentration of an unexposed wall between two exposed regions with acid concentrations of 100% after 20 minutes as a function of diffusivity. The room temperature diffusion rate for acid in SU-8 is typically about $1\text{x}10^{-16}$ m^2/s [7]. Although this number is too low for diffusion to occur, post bake heating will increase the diffusivity to the values seen and allow for diffusion to occur. In other resins it has been shown that in the short range of 20 °C, when the glass transition temperature is included in this range, a change of three orders of magnitude can occur in diffusivity [13]. Typically the top to bottom dose ratios increase the top dose to a value where it can be ten times larger then the bottom dose. So a concentration of 10% of the upper dose could give the same affect as the dose produced at the bottom of the SU-8. The second graph (Figure 3) shows that at the room temperature value of diffusivity the acid does not readily diffuse after a period of 72 hours.

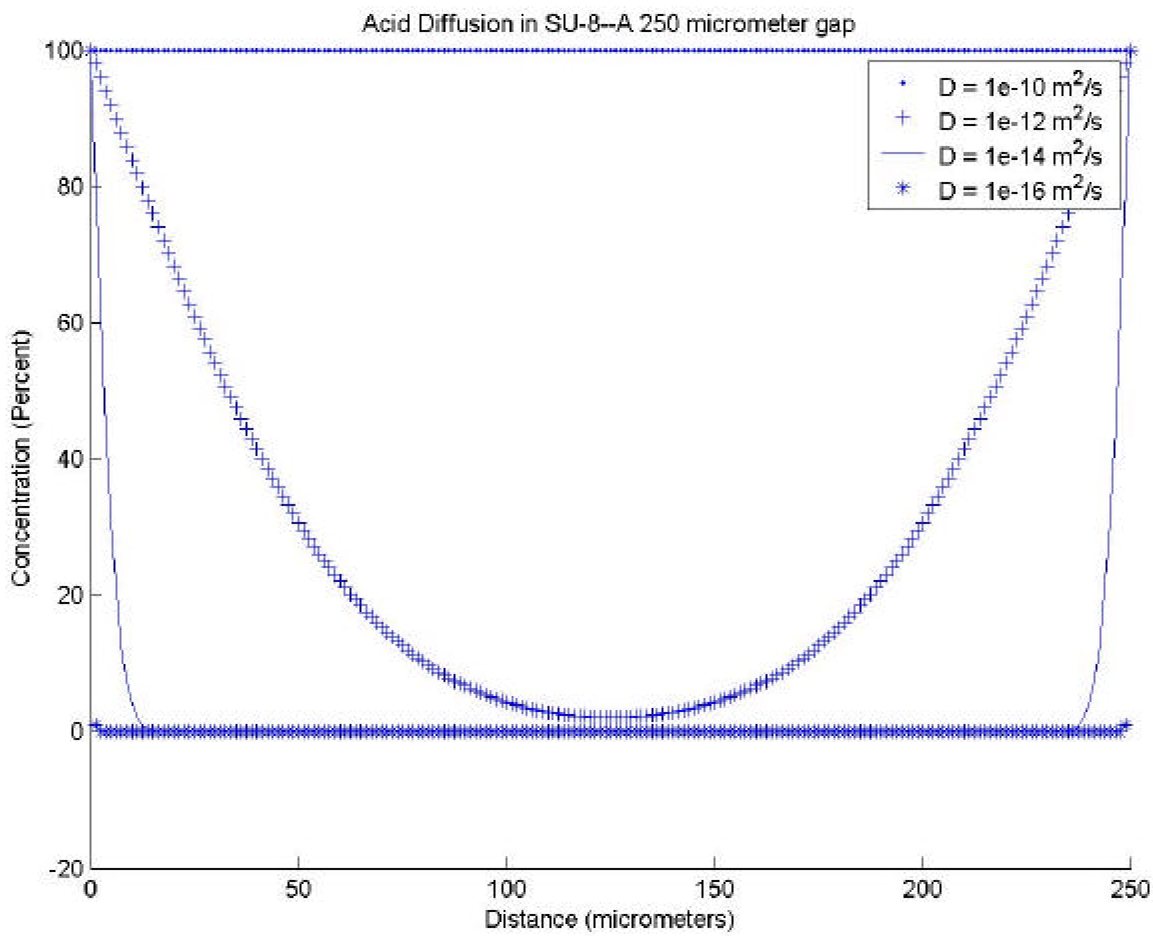


Figure 2: Plot of PAG concentration from simple model after 20 minutes between two exposed areas of SU-8.

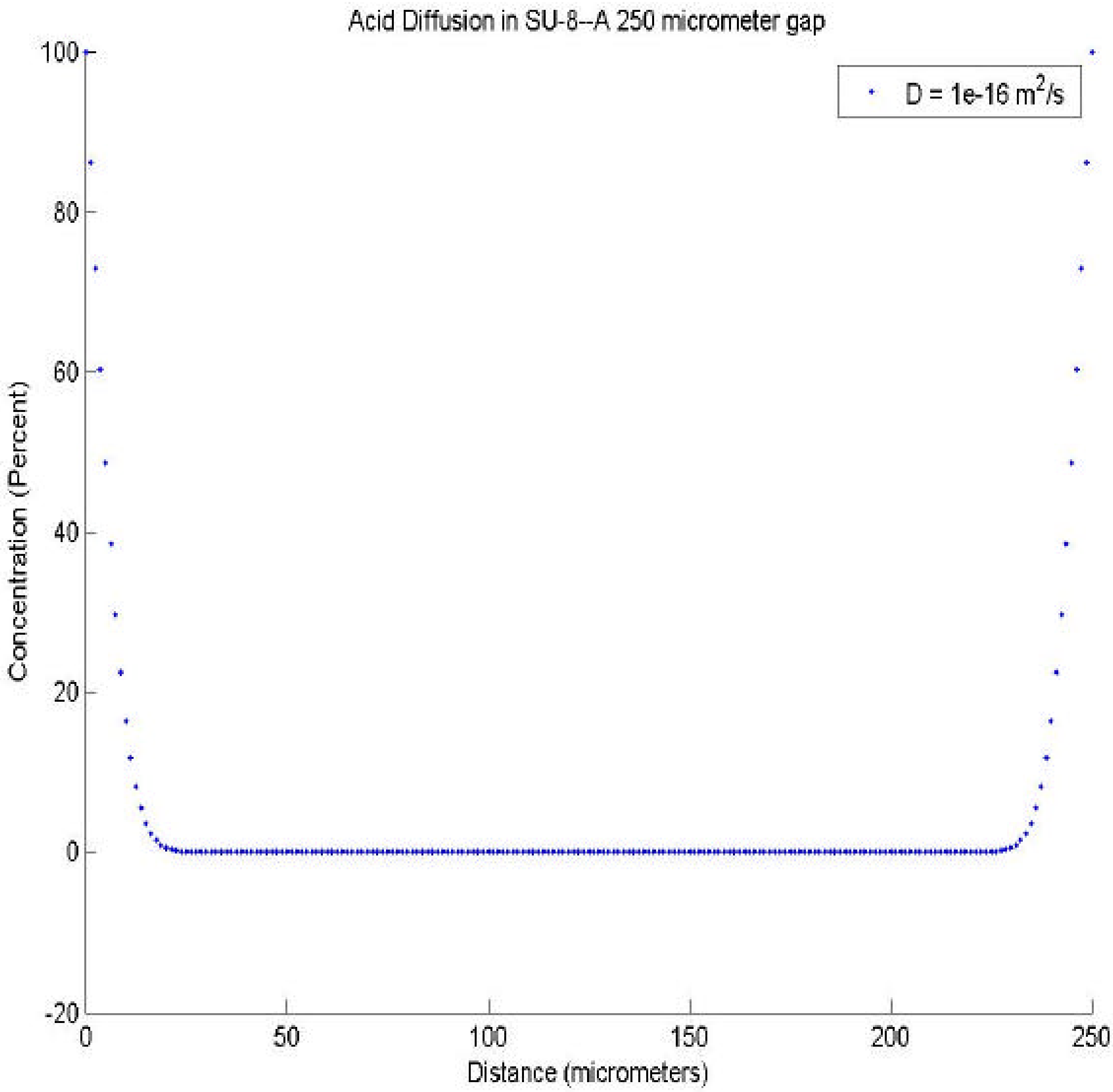


Figure 3: Plot of PAG concentration from simple model after 72 hours between two exposed areas of SU-8.

From these graph it is obvious that the diffusion of acid must be diminished. The goal of all the following experiments is to optimize the SU-8 manufacturing process in order to diminish the diffusion of acid. The parameters in each processing step that could effect diffusion will be discussed in this section. Table 2 below provides a synopsis of the discussion.

Table 2: Outline of SU-8 processing steps and the corresponding diffusion considerations for each step.

Process Step	Parameter that Affects Diffusion	Goal of Step for Diffusion Minimization
Pre Bake	Solvent Content	Reduce solvent content to lower diffusivity of acid through unexposed SU-8.
Exposure	Acid Concentration	Enough acid should be created to cross-link the SU-8 without making an excess of acid that will more readily diffuse.
Post Bake	Temperature/Time	The sample should be minimally heated to cross-link SU-8. High temperatures and bake times will increase diffusion.
Development	Mechanical Agitation	This does not directly affect diffusion, but it can be done if the previous steps were not optimal. The agitation can remove partially cross-linked SU-8.

It is believed that the amount of solvent in the sample must be controlled. Too little solvent may cause excessive stress. Also, for thick SU-8 samples, procedures are required to remove the solvent without over-baking the sample. When over baking occurs, the SU-8 begins to cross link and will be insoluble in the developer. However, solvent content levels in excess of the prescribed percentages are detrimental to the SU-8 in two ways. It may aid in the diffusion of the acid in SU-8. The more solvent in the sample, the less dense the cross-linking network, and potentially, the greater the diffusion rate. The diffusion will assist the acid in migrating into unexposed areas. In these regions there will be undeveloped SU-8 where there should be no remaining SU-8. The second affect is that bubbles will appear from the excess solvent and distort any small structures.

Exposing the sample is the next step in the process that was examined. From previous literature, it is unclear what the proper exposure values are. After completing the experiments on exposure parameters, it was found that too much exposure causes areas to cross link where no cross-linking should occur. The concentration of PAG is proportional to the dose. Therefore, high doses will create a large concentration gradient between the exposed and non-exposed regions, increasing the driving force for diffusion to occur. If the opposite is done, and the sample is under exposed, a different diffusion problem occurs. The exposed region will not be as dense as it should be due to a deficiency of cross linking. This might permit the developer to diffuse into the underexposed region, causing the SU-8 to swell and soften.

During the post-exposure bake, it is undesirable to overheat the sample and make the diffusion of the PAG increase. Also, if there is too little post-exposure baking then the sample will not properly cross link and it will be soft just as it would as if it was under-exposed. Both under-exposure and under-baking have the affect of under cross linking the SU-8.

The development process is relatively simple if all of the other steps were carried out properly. However, if there is a step that was not optimized, the sample must be submitted to agitation for long periods of time to mechanically clean out problematic areas. Otherwise, the sample only needs to be submitted to slight agitation in order to keep fresh development solution near the surface of the work piece.

The importance of optimizing process parameters to control diffusion depends upon the thickness and geometry of the SU-8 features. Exposures involving tall features (greater than 2 mm) with narrow unexposed regions between large exposed regions will amplify the adverse affects of diffusion. Densely packed features typically have small diffusion length scales and large exposed areas provide a significant reservoir of acid available for diffusion. In the graphs that are presented below it can be seen the difficulties that arise from densely packed features. The concentration profiles significantly shift in the negative y direction as the gap distance increases. It is shown in the below graphs (Figure 4, 5, and 6) that as the diffusion rates increase the diffusion of the acids can be sufficient to cover smaller gaps between exposed regions. As this distance becomes larger the acid cannot diffuse the entire span between exposed regions. This clearly demonstrates the amplification of acid diffusion as the density of the pattern increases.

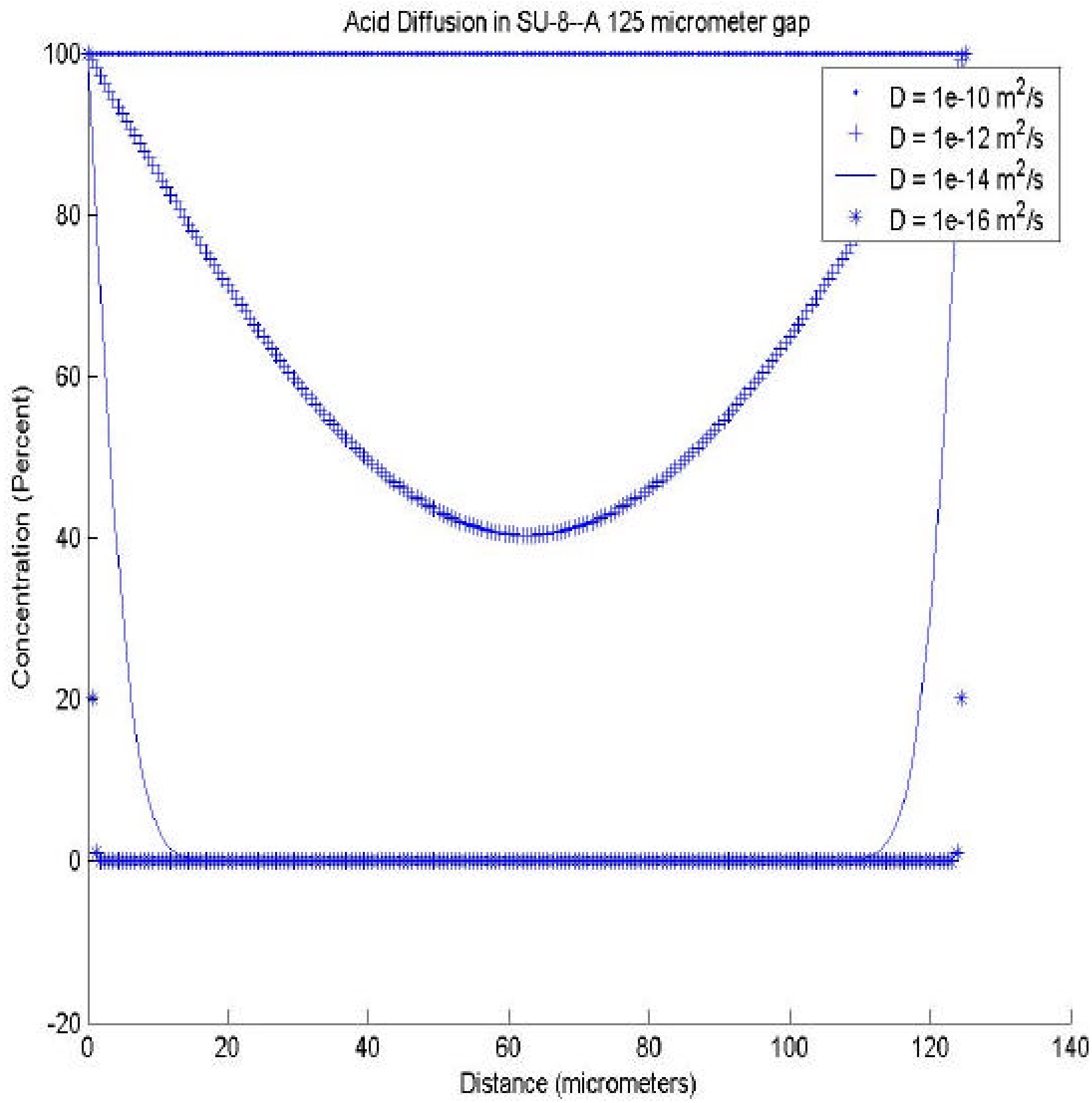


Figure 4: PAG diffusion model for 125 mm unexposed gap between two exposed regions. The diffusion time is 20 minutes.

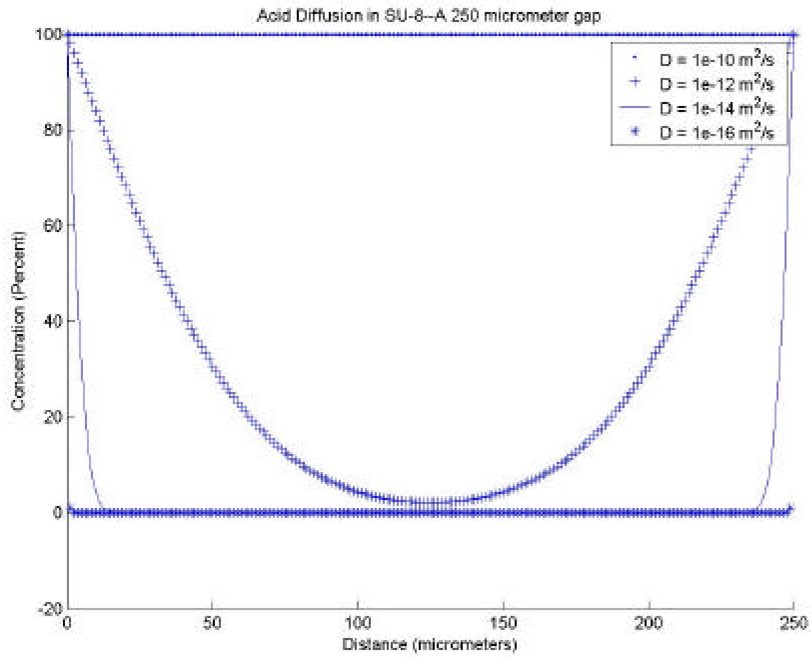


Figure 5: PAG diffusion model for 250 μm unexposed gap between two exposed regions. The diffusion time is 20 minutes.

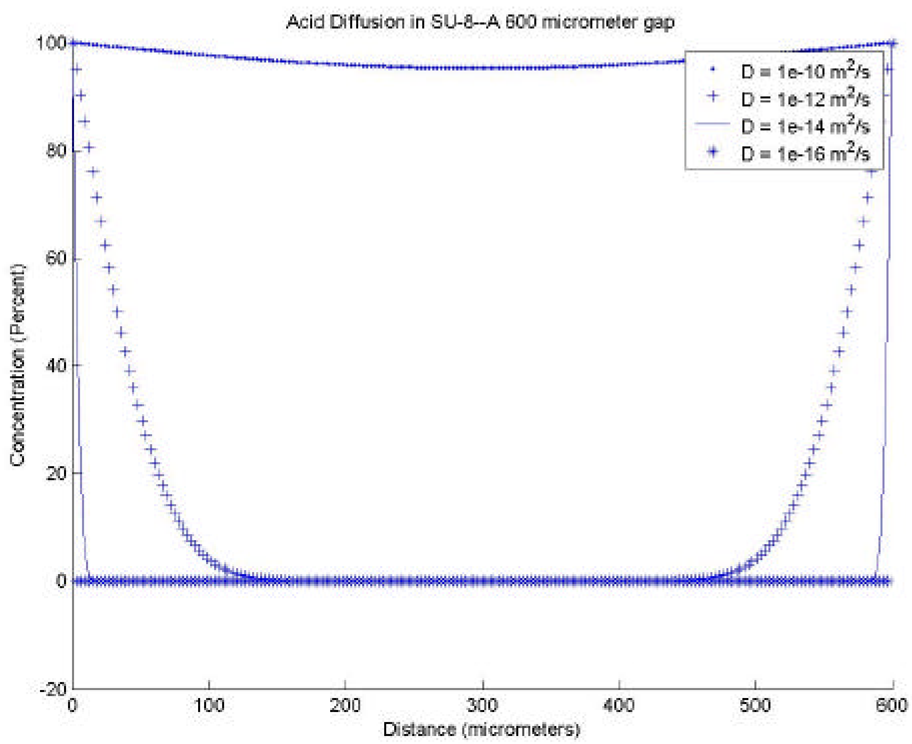


Figure 6: PAG diffusion model for 600 μm unexposed gap between exposed regions. The diffusion time is 20 minutes.

Substrate cleaning is not a parameter that affects acid diffusion, but it is still a critical step. If there are any trace amounts of acid in the SU-8 baking environment or on the actual substrate, the sample will be ruined. Small amounts of acid cause the SU-8 to cross link anywhere acids are present. This will make SU-8 x-ray lithography impossible.

In the past at LSU, it has been possible to make some SU-8 devices without optimizing the manufacturing procedures. These were samples with sparse patterns and short structures. This is advantageous because it minimizes the above-mentioned diffusion related problems. In thinner castings, it is easier to get rid of solvent because of the short diffusion lengths that the solvent must travel to exit the SU-8. While in the thicker samples, this is a problem due to the long diffusion distance that the solvent must travel to exit the SU-8. The other major problem that was avoided was diffusion of the PAG. This was done by having sparse patterns and by reducing the amount of solvent. When SU-8 is patterned far apart, the PAG is diffusing into an almost infinite sink. In the case where the patterns are tightly spaced, the PAG diffuses into the gap and creates a cap over the opening. This makes this area undevelopable.

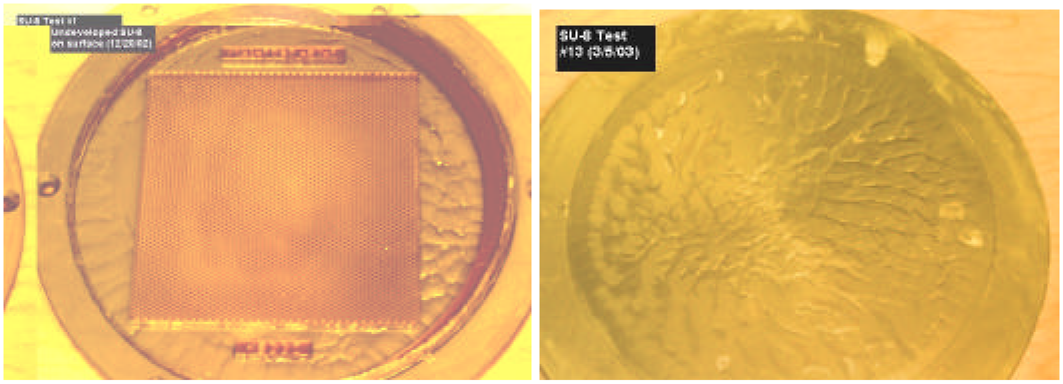
Finally, when the cast height was less than around 1.5 mm, it is relatively easy to simultaneously provide adequate dose at the resist/substrate interface while limiting the top-to-bottom dose ratio to an acceptable, low value. The thicker the SU-8, the more difficult it becomes to simultaneously provide adequate bottom dose and a low top dose.

2. SU-8 Experiments and Resulting Procedures

The following section is a collection of numerous experiments completed on SU-8 processing. These experiments will encompass all of the procedures comprising the fabrication of micro parts using SU-8. Results from experiments concerning each of the procedure steps will be presented along with the conclusions gained from every set of tests. Finally, the resulting procedure that will limit diffusion and produce the best results will be presented.

2.1 Substrate

The substrates that are used by the LSU Micro Fabrication Group are stainless steel discs 4.6875” in diameter and 0.375” thick. Stainless steel is used because it is conductive for electroplating, has a low reactivity, has good stiffness, and is relatively cheap. A lathe is used to flatten the surfaces of the stainless steel discs. Next, the surface is cleaned and roughened by sand blasting the surface with high velocity micro glass beads. The glass beads roughen the surface in order to create a stronger bond between the stainless steel and SU-8. The plate is then cleaned with soap, acetone, isopropanol (IPA), and deionized water (DI). Cleaning is preformed to ensure no chemicals or particulates remain on the surface of the substrate. However, cleaning was not sufficient to keep the stainless steel from being acid free. As stated previously, if any acid is on the substrate this will cause the SU-8 to cross link. In the early stages of casting SU-8, it was found that a thin film of undevleopable SU-8 was being formed on the surface of the stainless steel (Figure 7). This layer was found to be present even when no exposure was done. This indicated that the problem was occurring during the casting.



a) b)

Figure 7: Results when substrates were exposed to acid a) exposed sample with film on entire bottom surface b) sample that was never exposed that has undevlopable layer on surface of substrate.

Initially all of the variables of the casting procedure had to be tested. Through this process problematic variables could be eliminated from the casting procedure. From various experiments it was determined that the following parameters did not adversely affect casting: plastic cup material, type of PVC ring, uniform temperature profile (3° C variation was acceptable), humidity in room, type of soap used for cleaning, thickness of sample, drying of plate, sand particles being present, grease from the machine shop. The next set of experiments confirmed that there was an acid present in the manufacturing environment causing the undevelopable layer of SU-8. First, the substrates were immersed (prior to casting) in a solution of 50% NaOH to neutralize any acids that were on the substrate surface. SU-8 was then cast on the substrate. After baking, the sample was developed in SU-8 developer. The addition of the NaOH dip resulted in the SU-8 being completely developed. The next step to determine if an acid was present in the environment was to cast SU-8 in the clean room at The Center for Advanced Microstructures and Devices (CAMD) clean room facility. This is a highly controlled environment with no acids present (was currently doing SU-8 processing without the thin undevelopable film being present). To rule out any other source of this layer, all of the other parts of casting remained exactly the same. After casting at CAMD, the sample was directly developed; and all of the SU-8 was developed. Another CAMD casting was completed and exposed to x-rays and this resulted in a well-formed 2 mm sample. This showed that the environment of the laboratory was contaminated with acid. Further experiments proved that the environment of the LSU laboratory was the source of acid. The fume hood where the castings were created was cleaned with a commercially available acid cleaner, Citranox. A casting was then attempted in the freshly cleaned fume hood and this was a success as well. Figure 8 shows pictures of these samples and the resulting clean surfaces.

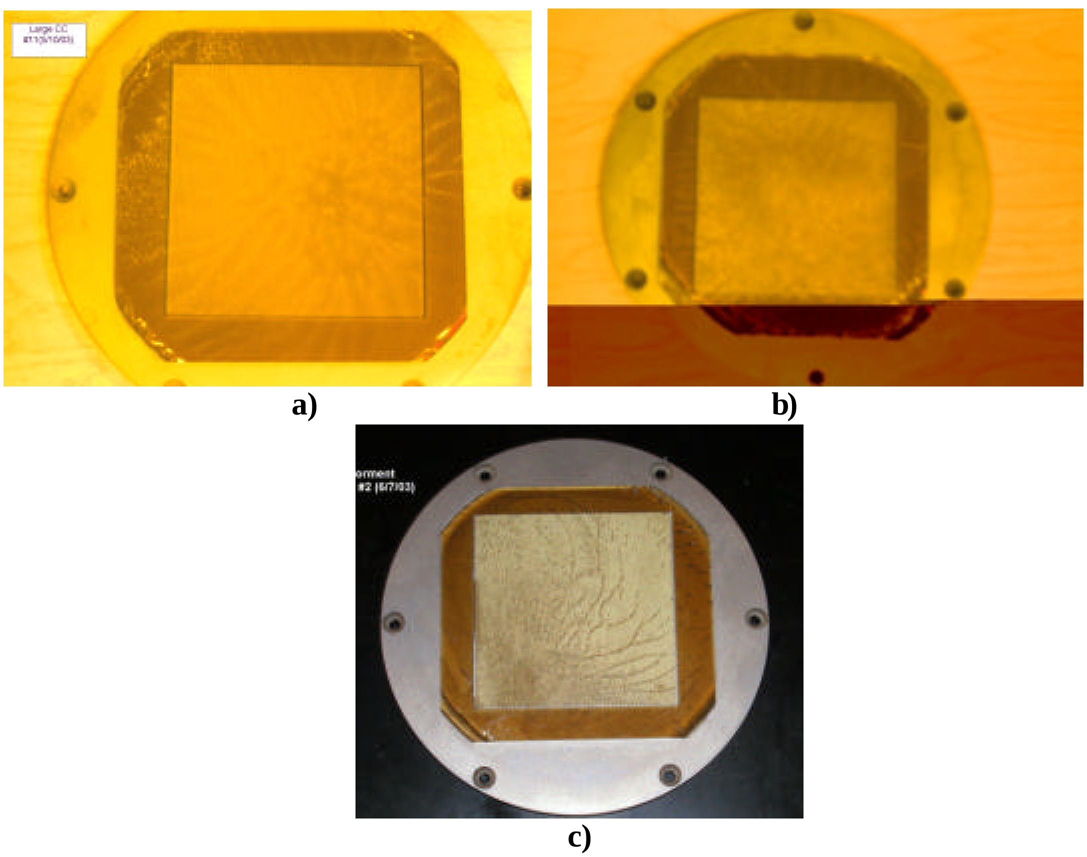


Figure 8: Three plates that had no cross-linked SU-8 resulting from acid exposure a) Plate soaked in 50% NaOH b) casting done in clean none acid environment (CAMD) c) casting done after fume hood was cleaned with acid cleaner

The source of the contaminating acid is believed to originate from the electroplating baths. The electroplating baths create hydrogen gas during active plating. This hydrogen gets into the air of the laboratory and deposits on any exposed surfaces. The conclusion was that casting could not be done in the μSET environment, nor could plates be stored here due to the exposure to hydrogen that inevitably resulted in an undevelopable SU-8 film present on the substrate surface.

2.1.1 Substrate Procedure

Since the source of the acid could not be eliminated from the micro fabrication lab, it was necessary to change the casting location. The castings would now be done in the constant clean environment of CAMD's clean room. The rest of the stainless steel substrate preparation procedure would remain unchanged. The plates would still be turned flat on the lath and then sand blasted. Next, the plates are cleaned with soap, acetone, IPA, and then DI. After cleaning, the stainless steel must be dried to remove any excess water. The plates are now ready for casting SU-8. The changed portion of the substrate preparation is the location of the casting and plate storage. It is now in an acid free environment. A very important key to successful substrate preparation is not exposing them to any source of acid. In the LIGA process, many laboratories have electroplating equipment that will produce hydrogen. To avoid this problem, no SU-8 manufacturing procedures should be done in the same environment as electroplating.

2.2 Casting SU-8

There are two choices when applying SU-8 to a substrate surface. The first is to spin coat a thin layer that has uniform thickness. This is good for shorter layers where uniform height is desired. The other method is to cast SU-8 on the substrate by confining the region that the SU-8 can flow onto.

The SU-8 selected for these experiments was SU-8 2075 [11]. SU-8 2000 series is an improved formula of the original SU-8 supplied by Microchem. When the SU-8 is received from the manufacturer it has a solvent content of ~26%. The solvent content, as stated before, should be around 7% for optimal results. This means that a reduction of the solvent content must be accomplished. A baking procedure is employed to reduce the solvent to the acceptable level. To achieve accurate solvent reduction the temperature and duration of the bake must be determined.

There are different time and temperature combinations that will result in the desired solvent content. If the proper combinations of time and temperature are not used one of two negative results will occur: over baking or under baking. Over baking is seen when the sample is heated for too long and/or at too high of a temperature. If this occurs, the sample will be prematurely cross linked and will be undevelopable even if no exposure is done. The opposite of this would be to under bake the SU-8. When this is done, not enough solvent is released from the sample. Two negative manifestations of excessive solvent concentrations is the fact that it has been empirically observed that bubbles form in high solvent content and “soft” SU-8 during the postbake after exposure. In addition, it is also possible that diffusion rates of PAG through the SU-8 (exposed or unexposed) is a function of solvent content. A central premise of this thesis is that diffusion of PAG from regions exposed into regions unexposed is the root cause of most problems associated with defining SU-8 features. So a higher solvent content could adversely affect the ability to confine the PAG to exposed regions.

The governing idea of pre-exposure baking is to get the solvent concentration down to ~7%. It discovered that when a thick sample was cast using a single pour casting method, it was impossible to reduce the solvent levels to the desired values. High levels of solvent resulted in two outcomes that hurt the SU-8 manufacturing process. The high solvent content seemed to result in the formation of bubbles causing distortion in the shape and integrity of the posts, as shown in Figure 9 which is a 2.7 mm thick sample exposed with a bottom dose of 10 J/cm^3 at CAMD XRLM1 using an additional filtering of 61 µm aluminum resulting in a TBR of 8.17 and the posts are 480 µm from flat to flat and the spacing between the posts is 125 µm. This sample was post baked for one hour at 50 °C and developed for 3.5 hours. Also, it appeared that the extra solvent made the PAG more readily diffuse. The solvent could increase diffusion rates of the PAG by decreasing the material density and by giving the PAG a path of less resistance to travel through because of the decrease in hardness in the sample, which increased the diffusivity of the PAG. Not only did the solvent decrease density, it also enabled the PAG to have a path of low resistance for diffusion. When the PAG diffuses more readily this will result in non-exposed areas having acids in them, cross linking the SU-8. The end result will be undevelopable parts of the sample that should have been developed. Figure 10 shows two patterns that have diffusion of PAG into unexposed regions. The picture on the left is a 4 mm thick SU-8 sample with holes 500 µm from flat to flat and spaced 420 µm apart. On the right is 2 mm exposure with posts that are 480 µm from flat to flat and the spacing between the posts is 125 µm. Both sample were post baked for one hour at 50 °C and developed over night.

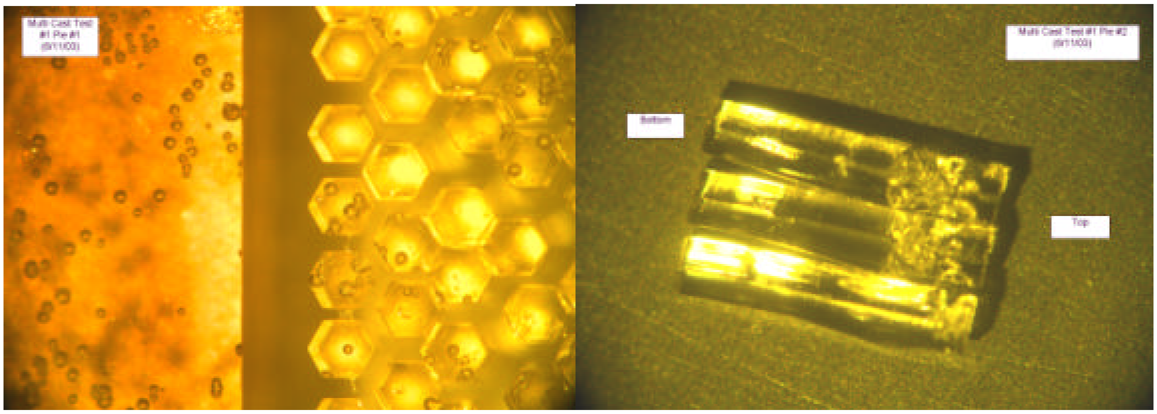


Figure 9: Bubble formation in SU-8 from excessive solvent.

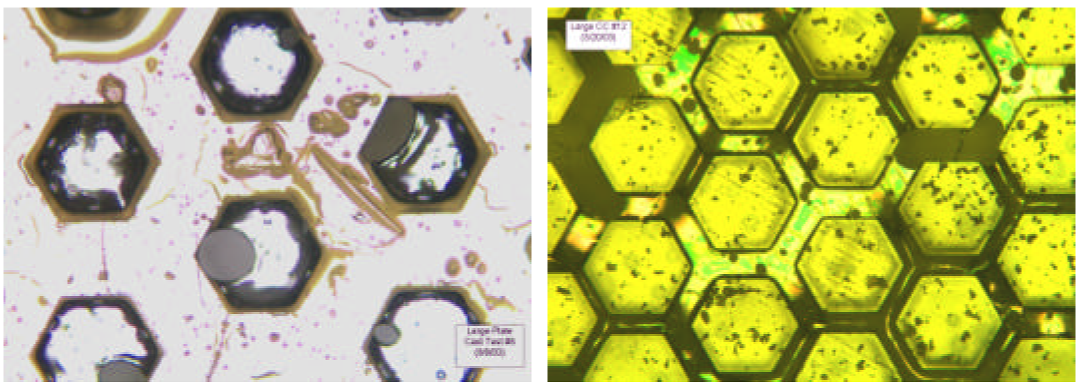


Figure 10: Unexposed regions with undevelopable SU-8.

Initially, the set up casting procedure was a method developed previously at LSU by Jian Zhang (which will be referred to as the single pour cast). An in-house heating apparatus was created to do the castings. The procedure was [16]:

1. After cleaning substrate, a PVC ring is clamped to the surface of the substrate. The casting surface should be then leveled to get a flat SU-8 surface.
2. Pour a measured volume of SU-8 onto the stainless steel surface. The volume was determined by the required height and the corresponding values are shown in Table 3.

Table 3: Old relationship between poured SU-8 and created height [15]

Volume of SU-8 Poured	Final Height of SU-8 (before machining)
mL	mm
10	0.7
15	1.05
20	1.4
25	1.76
30	2.11

3. Turn hot plate to 105 °C and turn heaters on. Cover the sample with glass plate and aluminum foil to prevent particle and light exposure.
4. Baked for the required time given in Table 4.

Table 4: Old required bake times

Final Height of SU-8 (before machining)	Time of Bake
mm	Hours
Less than 0.5	6
0.5 to 1.0	18
1.0 to 1.5	24
1.5 to 2.5	30

5. Turn off hot plate and allow sample to cool to room temperature. All handling of SU-8 should be done under non-UV light due to high sensitivity.
6. The sample is then fly cut to desired final height. This is done to obtain a flatter surface.

Experiments were conducted using this method and it was found that the samples had greater then 12% solvent remaining in the samples. The next experiment done was a multiple pour casting in order to give the solvent a shorter distance to exit the SU-8. A small amount of SU-8 would be poured into the cast and then baked properly. The following layer was cast directly on top of the previous layer. After this layer was baked to the proper specifications, the procedure could be repeated until the desired height was reached. The execution of this experiment resulted in an interesting discovery. As each layer was added, the SU-8 was weighed and the total amount of solvent remaining was determined. It was found that the first layer was the only layer that had the desired amount of solvent remaining. As subsequent layers were added, the amount of solvent remaining slowly increased. The more layers that were added made it harder to reach the proper solvent content. Table 5 has the values that were gained as the experiment progressed. The mechanism that was governing this was the diffusion of the solvent.

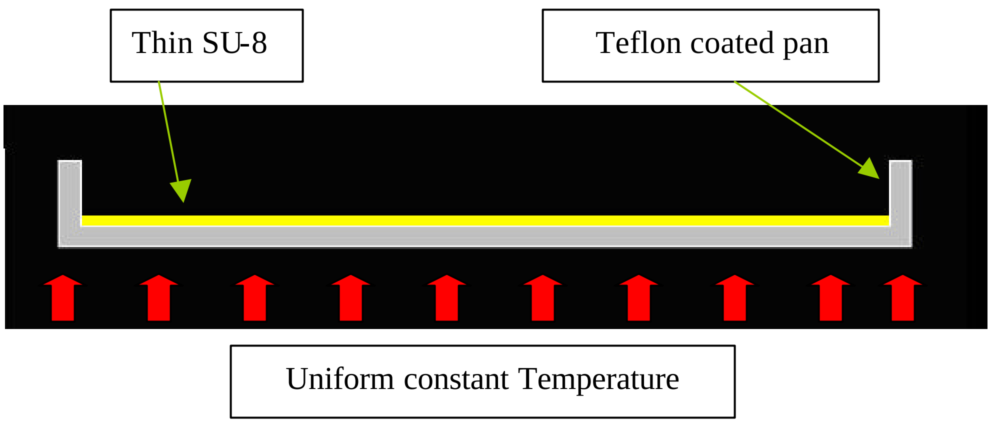
Table 5: Results of Multi Cast Test Experiment.

Multi Cast Test					
Layer	Volume Poured	Bake Temp.	Bake Time	Solvent in Layer	Total Solvent
	ML	Degrees C	hours		
1	10	105	10	4%	4%
2	10	105	10	13%	8%
3	10	105	10	15%	11%
Additional Bake	NA	105	5	NA	10%
Additional Bake	NA	105	5	NA	10%
Additional Bake	NA	105	5	NA	9%

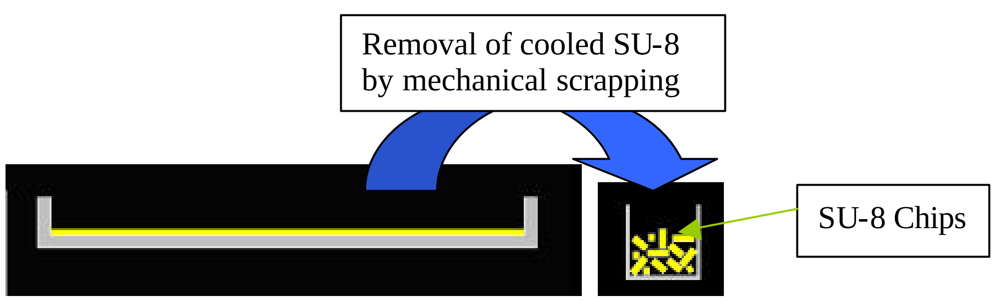
The first layer was thin and initially had uniform solvent. With the addition of heat, it was possible to drive out the remaining solvent in the initial layer. However, in the subsequent layers the solvent was not only driven out, but it went down into the lower layers that had less solvent present, due to their previous baking. After the SU-8 diffused downward and equilibrated, the diffusion lengths became too long for the solvent to exit the sample without over baking.

2.2.1 Casting Procedure

The first layer cast was the ideal SU-8 sample due to its solvent content and the uniformity of the solvent content. The solvent content had to be fairly uniform because of the small thickness of the sample did not allow for any significant gradient to be present. Since the best SU-8 was the first layer cast, it was decided to make the entire SU-8 sample from a thin cast layer. The basic idea was to cast a thin layer of SU-8, then take this thin layer and recast it into a thick SU-8 sample. The resulting chip casting procedure is given in Figure 11:

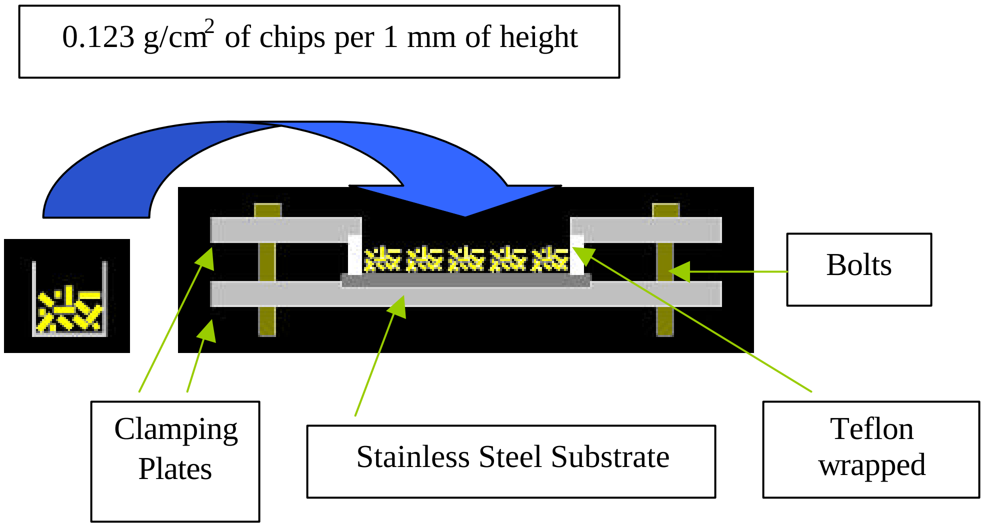


1. Thin layer of SU-8 solvent bake: The SU-8 is put on a hot plate making the Teflon coated pan's temperature 101 °C for ten hours. Pour volume is 1.25 mL per square inch.

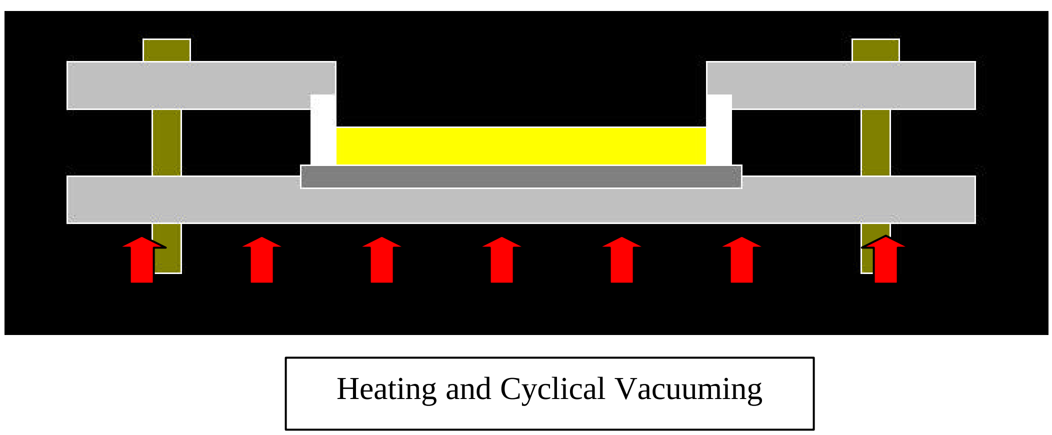


2. Chip removal: After cooling to room temperature the SU-8 is mechanically removed with a plastic spatula to create small SU-8 chips.

Figure 11: SU-8 casting procedure. (figure continued)



3. Substrate Casting Set-up: Teflon tape wrapped PVC ring is clamped to a stainless steel substrate and the SU-8 chips are place in the center. For each millimeter of height desired place 0.123 g/cm^2 of SU-8 chips into the jig.



4. SU-8 Puck Creation: The set-up casting apparatus is placed in a vacuum oven at a temperature of 105 °C. Cyclical vacuuming is done until no bubbles remain in the SU-8.

(figure continued)

Thick SU-8 Ready for Exposure

5. Final Product: When the Teflon wrapped PVC ring is removed, the SU-8 is fly cut to the final height. The thick SU-8 sample is then ready for exposure.

1. Solvent Releasing Cast: The solvent-releasing cast will be done in a Teflon coated aluminum pan. SU-8 does not adhere to the Teflon coated surface. This will make the removal for the shape casting easier. The solvent-release cast will be implemented with parameters that mimic the first layer of the multi-cast test in order to obtain the same results. The area of the new casting is 8 times larger than the initial test (old area was a 4” diameter circle and the new area is a 10” square). This means that pour volume will be 80mL to get the same thickness of SU-8. This equates to pour of 0.124 mL/cm^2.
2. Baking: After the SU-8 is poured into the Teflon pan, the sample is then heated for ten hours. Because of the previous acid problems the old custom made heating jig will no longer be used. The pan will be placed onto a temperature-controlled hot plate, located in CAMD’s clean room, which is covered with one layer of aluminum foil for surface protection. To get the same surface temperature profile as before, the hot plate must be set to 125 °C. This was found by taking the average surface temperature, 101 °C, of the stainless steel substrate used in the old casting jig under casting conditions. The temperature setting

found for the hot plate matches this average surface temperature. **It should be noted here that the chips should be hard and brittle NOT soft and bendable. If the chips ARE soft, then the baking was not sufficient. This main cause of this is uniform thickness of SU-8 and uniform temperature of baking surface. One can reduce the poured volume and increase time to offset this problem.**

3. Chip Formation: Allowing the sample to cool to room temperature follows heating. The SU-8 is then mechanically removed from the Teflon surface. This is done using a plastic spatula, so the Teflon coating is not damaged. The hard SU-8 chips that are formed should be placed in a temporary holding container.
4. Shape Casting: The next step is to cast the SU-8 on the stainless steel plate to the desired thickness. A PVC ring is wrapped in Teflon tape, so the ring may be removed more easily from the SU-8 after casting. This ring will shape the SU-8 chips into a solid "puck" of SU-8 on the stainless steel substrate. The ring is then clamped to the surface of the substrate. SU-8 chips are placed onto the substrate within the area surrounded by the PVC ring. The amount of chips depends on desired thickness of the end sample. For each millimeter of height desired, 0.123 g/cm^2 of chips should be used. In the case of a four-inch circle puck, the result is 10 grams per millimeter of height. The entire setup is then placed into a vacuum oven heated to 105 °C. The heat melts the chips, while the vacuum is needed to remove the air bubbles. The sample should be vacuumed before heating is begun to reduce air bubbles. As soon as the sample is placed into the oven and heated, the SU-8 should be cyclically vacuumed until the SU-8 has no remaining air

bubbles and is a solid puck. Again leveling of the oven must be completed to get a level sample.

5. Final Product*:* The final step is to let the sample cool to room temperature. Now the ring is removed by cutting the Teflon tape around the ring's perimeter. The ring is then pried away from the substrate's surface. Finally, the sample may be fly cut to the exact height desired.

This results in samples of any desired height with a uniform solvent content of ~7% solvent. Completion of solvent test on samples using a Thermo Gravimetric Analyzer showed that the solvent content is indeed uniform throughout the sample. The procedure also results in some other advantages. The bake time of a sample is cut down to 1/3 of the previous casting time because of the thin layer, and the sample is hard. In the past the samples have been soft, which resulted in more damage that can be caused to the sample during handling/exposures.

2.3 Exposures

Because SU-8 absorbs energy and acts as a filter for underlying SU-8, the dose absorbed in the SU-8 decreases monotonically from a maximum value at the top to a minimum at the bottom. The ratio between the amount of energy absorbed at the top to that absorbed at the bottom of the resist is called the top-to-bottom ratio (TBR). Figure 12 shows a typical dose profile for a 2.5 mm thick SU-8 sample with a "moderate" degree of filtering (200 μm-thick graphite mask membrane and 61 μm of aluminum foil). The TBR can be altered by the use of filters (aluminum foil of thickness varying from 20 to 300+ μm, depending upon the resist thickness) that preferentially absorbs softer radiation. Harder radiation creates a more uniform dose profile in the SU-8, reducing the TBR. In fact, with sufficient filtering it is possible to achieve a TBR that approaches unity. As will be discussed, it is virtually impossible to produce SU-8 features in a high-density pattern and with feature heights greatly exceeding 2.0 millimeters if the TBR exceeds a value of approximately three. However, achieving such a low TBR in a thick sample requires a higher energy spectrum (such as the "wiggler"), otherwise exposure times can be excessive (on the order of 10 hours or greater).

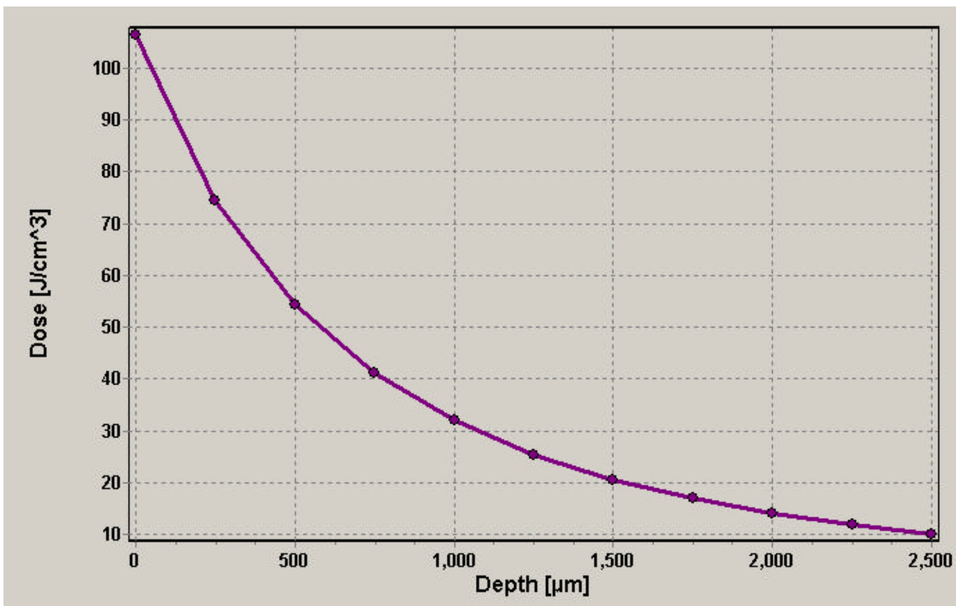


Figure 12: Typical dose profile for a 2.5 mm SU-8 sample exposure with a top to bottom ratio of 10.64 (Profile from CAMD XRLM1 using 61 μm aluminum filter).

It was discovered that as the top dose increased, the portions of the SU-8 that were intended to develop, did not develop, especially in dense patterns. As mentioned earlier, the top dose is the product of the bottom dose and the TBR. Furthermore, for a given filter, the TBR increases significantly with resist thickness. The PAG concentration within the SU-8 is proportional to the dose. Also, the transfer of PAG via diffusion into undeveloped regions is a function of the PAG concentration in the neighboring exposed SU-8, *and* the diffusion distance, *and* the diffusivity of the PAG within the SU-8.

When PAG does diffuse between neighboring exposed and unexposed regions beyond a critical threshold it becomes insoluble. Lateral diffusion of PAG into the undeveloped regions is always greatest near the surface of the SU-8. This results in “an insoluble cap” that spans the gap between neighboring exposed regions. Varying degrees of the “capping” phenomena are described in Figure 14 and 15.

Figure 13 shows a typical example of capping where the width of the feature greatly exceeds the desired value only over the top 10-20% of the feature height (where the dose in the exposed regions are highest). In this case, opening in the cap gives the developer a way to reach the developable SU-8 beneath. An unsupported bridge is then created that can more easily be broken by agitation of the developer. Figures 14 and 15 are schematics showing varying degrees of the “capping” phenomena, ranging from a complete inability to develop the sample to minimal “lips” at the tops of the structures that may be acceptable, depending upon the application.

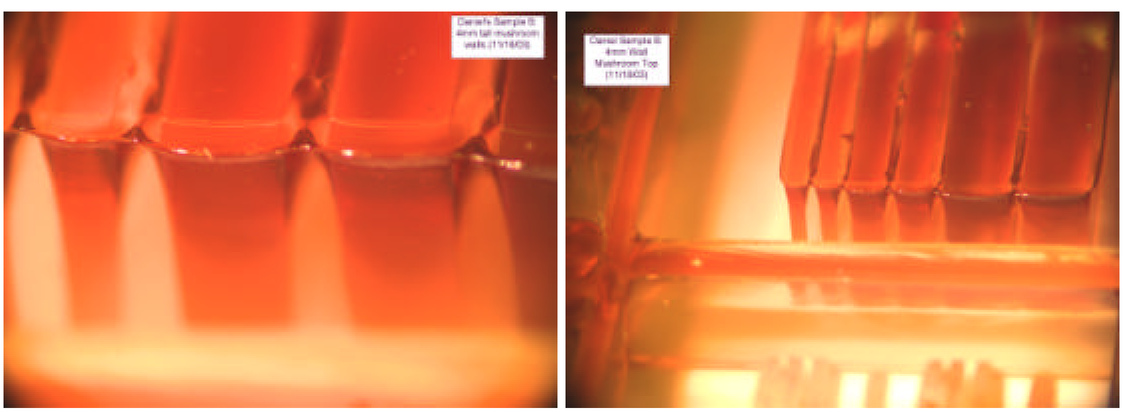
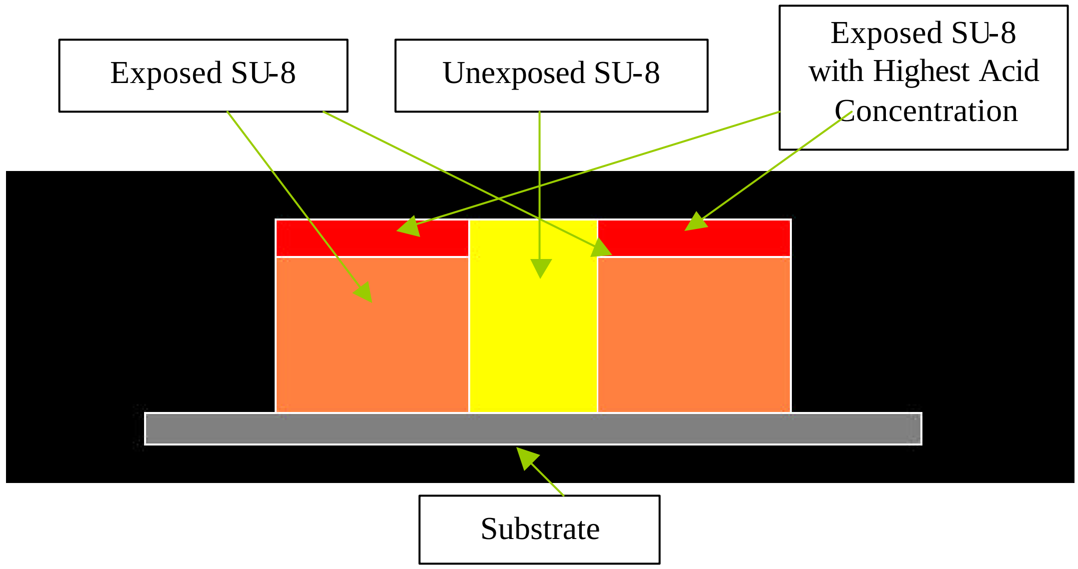
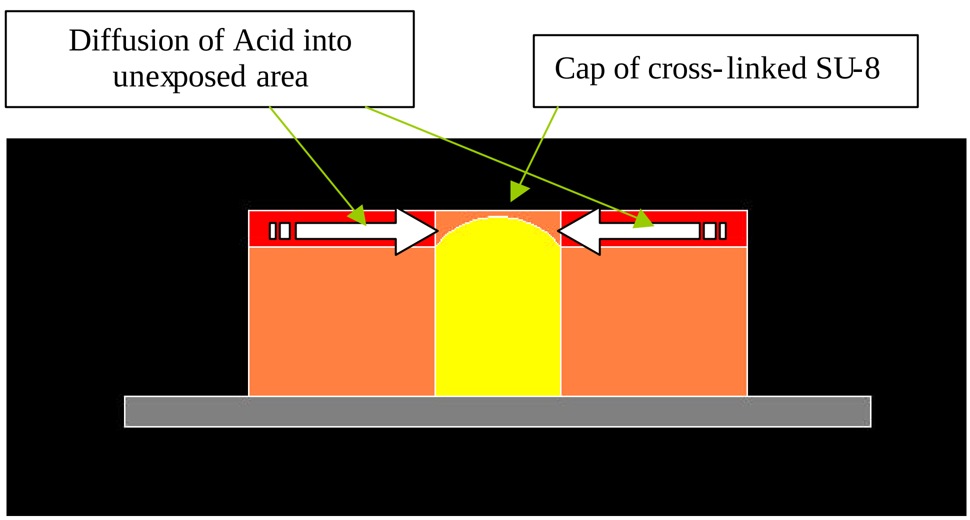


Figure 13 Example of a SU-8 sample with capping when top to bottom ration is to high.

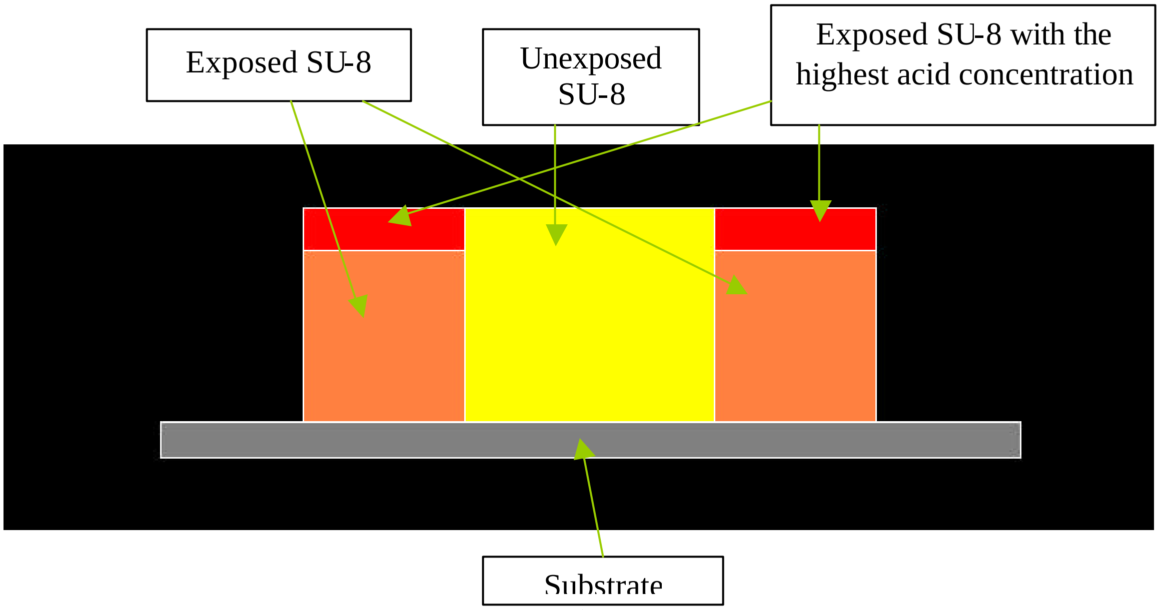


a)

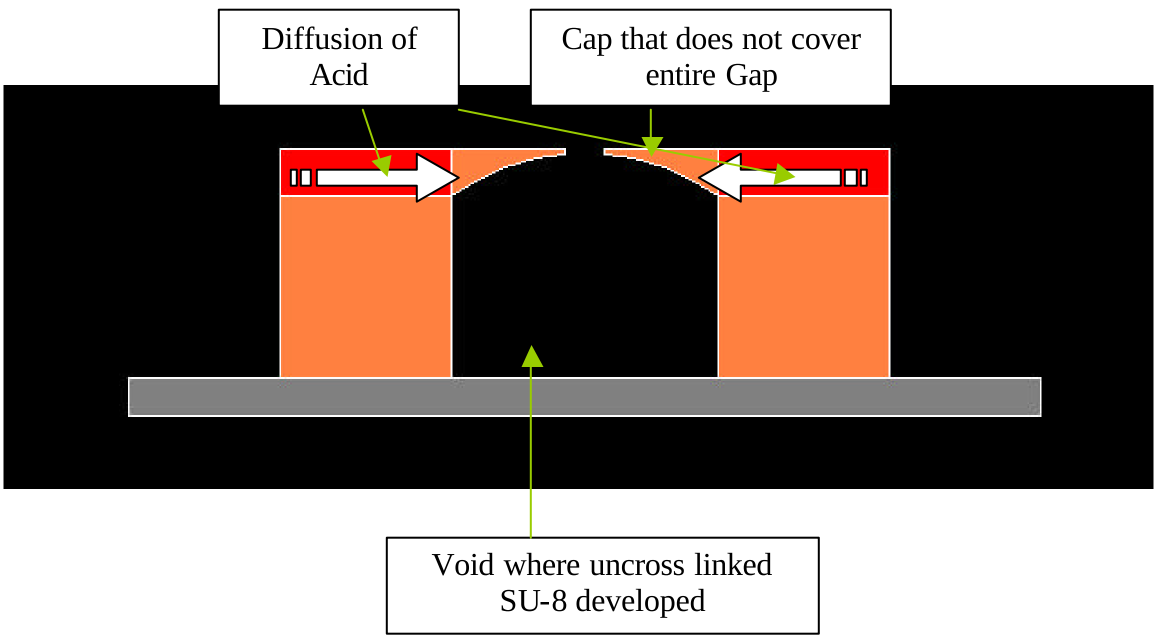


b)

Figure 14: Capping diagram a) Exposed SU-8 that is relatively close together (depends on acid concentration and distance). b) Diffusion of acid into unexposed region forms a cap of undevelopable SU-8. If the gap is close enough the cap will be strong enough to withstand agitation of development.

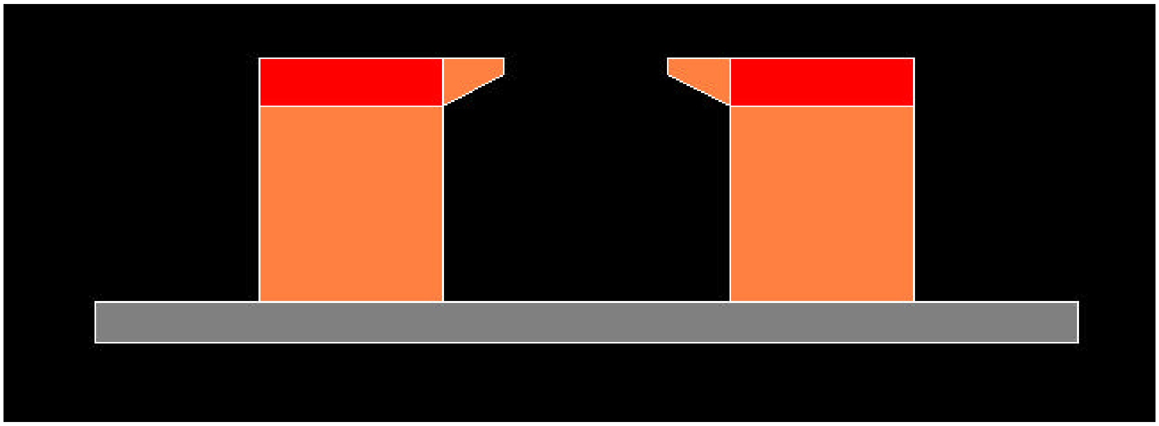


a)

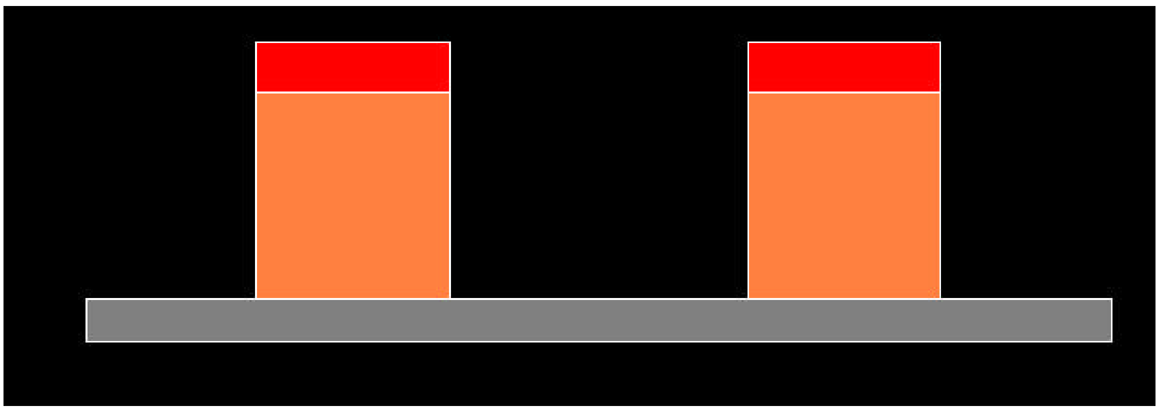


b)

Figure 15: Diagram of different types of caps a) Two exposed regions with a large gap between them b) Cap that does not cover entire gap between exposed regions. The non-exposed SU-8 will be developed due to the opening. The cap could stay an unsupported bridge or depending on strength and thickness it could c) break off into shorter bridge or d) completely break off from lack of support. (figure continued)

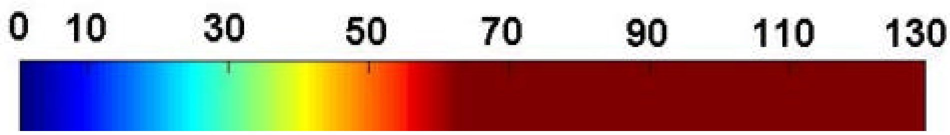


c)

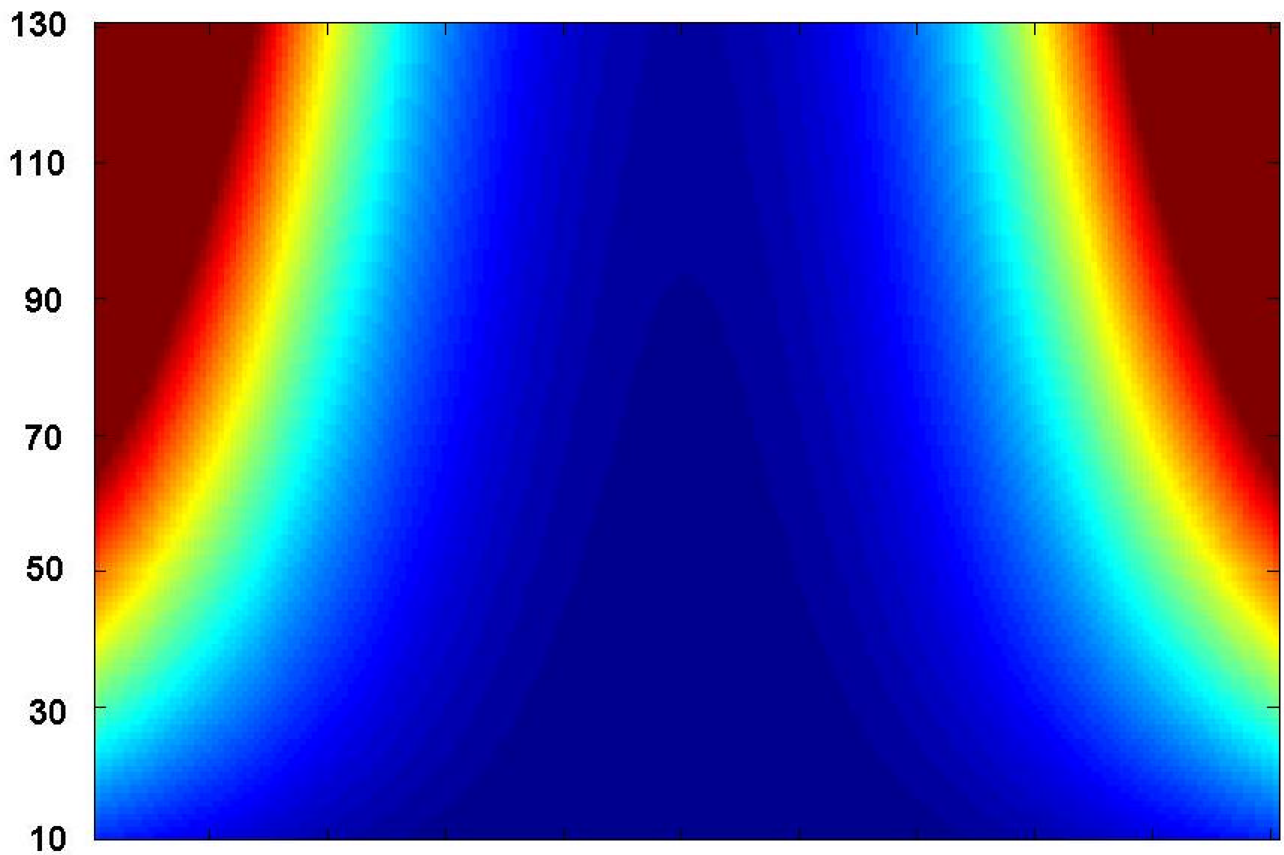


d)

A simple one-dimensional model has been built to determine if it is plausible for acid diffusion to be the cause of the observed results. The complexity of the actual physical process, as stated before, prohibits a simple diffusion model from giving precise results. It does however provide an order of magnitude prediction of the combined effects associated with length scale, diffusivity, and PAG concentration. The model does provide a qualitative prediction of the shape of the cap that is seen experimentally. Figure 16 provides a typical model prediction of concentration profiles within an infinite channel of unexposed SU-8. The dose within the exposed regions varies from a bottom dose of 10 J/cm^3 to a top dose of 130 J/cm^3. For given channel width (125 and 250 μm), and diffusivity (10^{-12} cm^2/sec), and a 20 minute post bake, a two dimensional concentration profile results within the channel where the higher dose regions would be unlikely to develop. The diffusivity value chosen is implicitly a strong function of the post bake temperature as stated in the introduction.

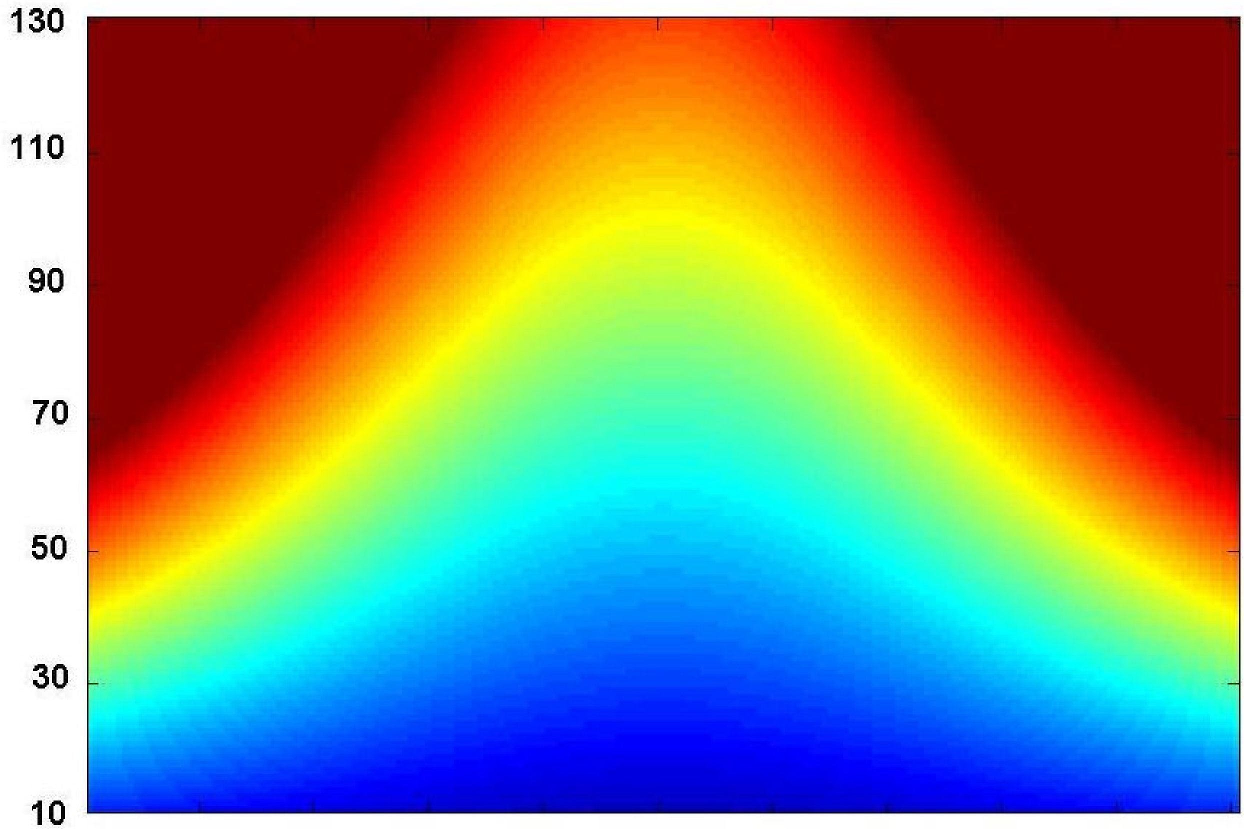


(c)



(b)

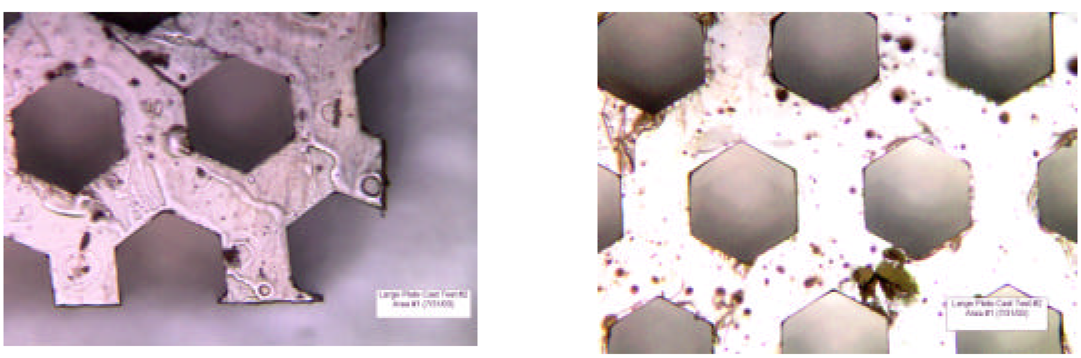
Figure 16: 2-D SU-8 capping model. These figures are the results of the diffusion model constructed. Here two results are presented with a diffusivity of $10e^{-12}$ m/s and time duration of 20 minutes. The figures represent unexposed SU-8 between two exposed regions of SU-8. The values on the left of the pictures are the dose values in the neighboring exposed SU-8. (a) gives the scale of color that corresponds to the appropriate dose. (b) is a 250 μm gap of unexposed SU-8 and (c) is a 125 μm gap of SU-8. (figure continued)



(c)

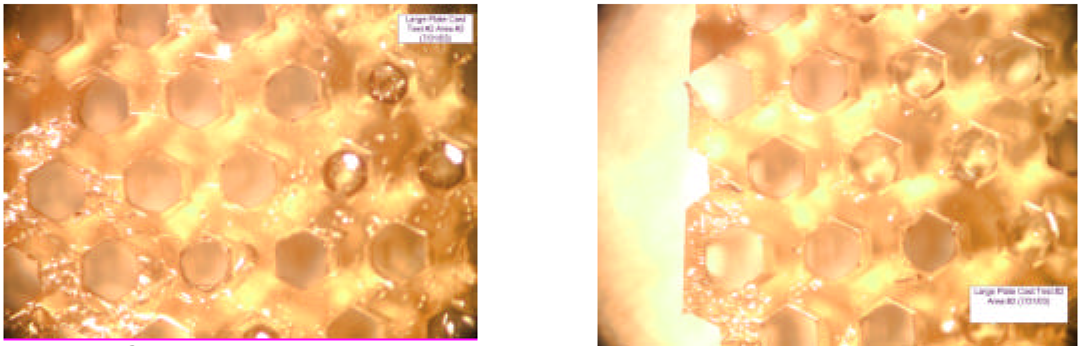
In the displayed model in Figure 16 one should note that the 125 μm gap has a cap form due to high levels of acid present across the upper portion of the unexposed SU-8 while in the 250 μm sample the diffusion is limited to form an incomplete "cap". This model even predicts a shape that is similar to Figure 15. Congruently, the figures show why a high-density pattern is the most difficult to successfully accomplish. It can be easily seen that in Figure 16, the gap of 125 μm has much higher concentrations of acid than the gap of 250 μm, using identical diffusivity and duration of diffusion. The diffusion rate used in this model is chosen only to display that adverse affects of diffusion and not as a true representation of actual diffusion. This model gives a good starting point to comprehend the adverse consequences of excessive diffusion during SU-8 processing.

Experiments were done that distinctly show the relationship between higher top dose and closing of gaps. The pictures of Figure 17 clearly demonstrate the slow closing of the gap between the exposed regions as the top dose increases. A series of exposures were performed on a single 2.5 mm thick SU-8 layer, varying only the bottom dose. The geometry of this sample is hexagon holes with a wall-to-wall diameter of 500 μm and a spacing of 420 μm. The post processing on this sample was 50 °C for one hour and developed 4 hours. Since the TBR was constant, the dose on the top of the SU-8 increased proportionally to the increase in the bottom dose. From the standpoint of reducing the "capping" phenomena, it was found that the minimum dose used in this experiment, 10 J/cm^3, produced the best results. As the bottom dose was increased (15 J/cm^3, 20 J/cm^3, and 25 J/cm^3), the volume of undeveloped SU-8 in unexposed regions significantly and monotonically increased.

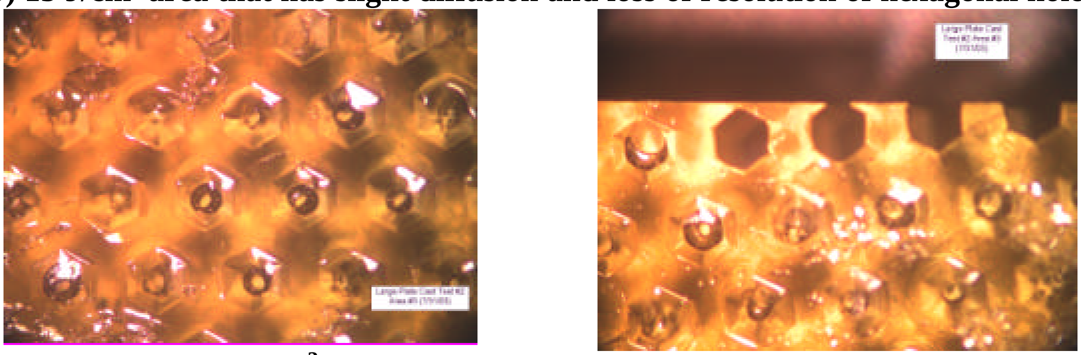


a) This is the 10 J/cm^3 with completely open holes.

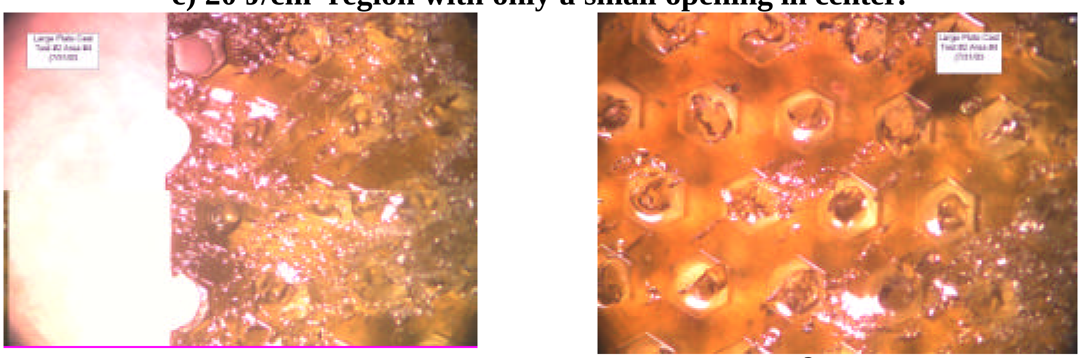
Figure 17: Exposure dose and diffusion relationship pictures. Shows increased dose of exposure increasing diffusion and closing of the holes. (figure continued)



b) 15 J/cm^3 area that has slight diffusion and loss of resolution of hexagonal hole.



c) 20 J/cm^3 region with only a small opening in center.



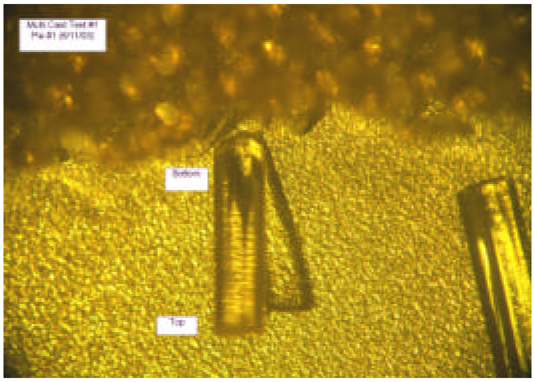
d) Complete closer of the holes in the 25 J/cm^3 case.

While a dose of 10 J/cm^3 was optimal from a standpoint of limiting unwanted diffusion effects near the surface, the dose was not sufficient to adequately cross link the SU-8 at the bottom. As a result, the SU-8 features were soft from underexposure. 10 J/cm^3 has been found to work the best because of the high top to bottom ratio. For this experiment, the TBR was 8.17 resulting from an exposure done at CAMD on XRLM with additional filtering of 61 μm of aluminum. Therefore, for this geometry/ post bake combination (50 °C for 20 minutes), a top dose of around 81.7 proved to be the upper limit that would acceptably minimize the adverse effects of diffusion. Even at the next lowest dose (120 J/cm^3), significant problems begin to be observed.

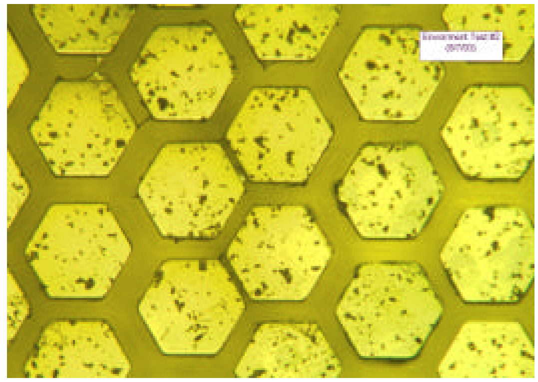
Underexposure is just as problematic. When the dose is not sufficiently high, the cross linking density and accompanying stiffness is reduced. When immersed in developer, underexposed SU-8 swells and softens, presumably because it is absorbing developer. The swelling does not occur in regions where the SU-8 is cross linked to a higher degree. For taller structures the necessary agitation done during development will cause the soft areas to be a structurally weak as demonstrated in Figure 18. The structural instability shown in Figure 18 comes from a 2.5 mm sample done on XRLM1 with 61 μm aluminum filtering resulting in an 8.17 TBR for the geometry of posts that are 480 μm from flat to flat and with a spacing of 125 μm. The sample was post baked for one hour at 50 °C and developed for 3.5 hours. This weakness can cause the SU-8 to loose adhesion to itself and cause the upper portion of SU-8 to come off of the substrate. It may also cause the bending of structures due to the softness. The underexposed SU-8 is best seen in small individual exposed SU-8 structures. In larger exposed SU-8 structures, the diffusion of acid from other parts of that region and the small amount of surface area/volume for developer to enter, aids in it remaining solid. Figures 19 and 20 pictorially display the differences between diffusion of developer into a large exposed area and a smaller exposed area.

2.4 Post Exposure Bake

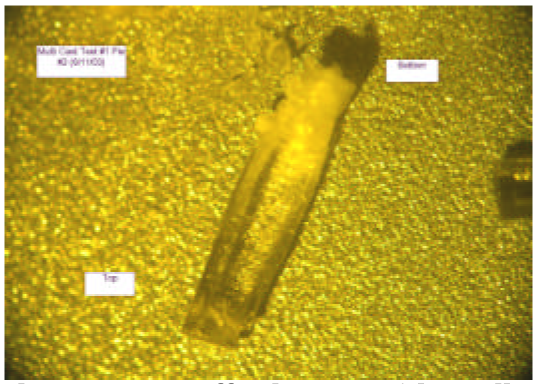
Post-exposure bake is necessary to cross link the SU-8, but too much baking promotes diffusion of acid into the unexposed regions. Diffusion occurs more readily as



a) A bent post that is still attached to substrate.



b) Top view of post that have deflected because of soft SU-8.



c) A post that was torn off substrate with swollen bottom.

Figure 18: Results of Diffusion of Developer into Exposed SU-8.

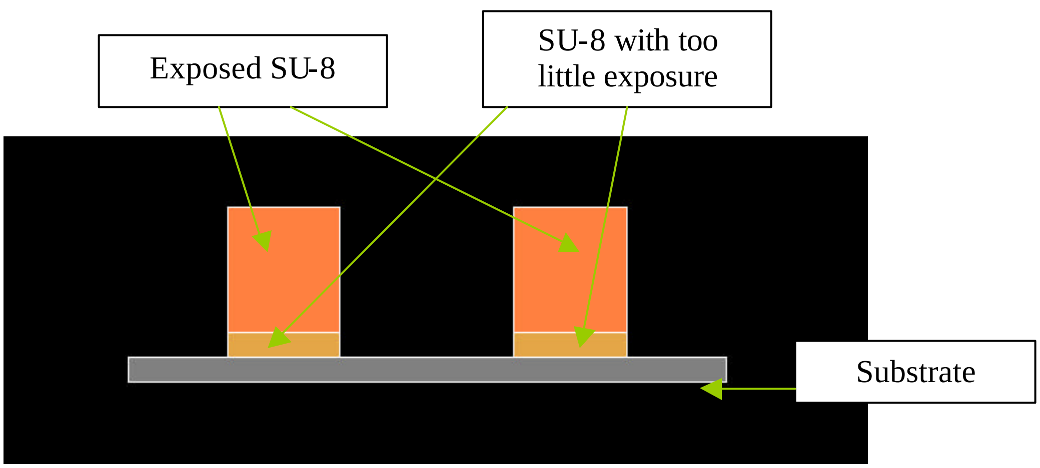


Figure 19: Diagram of underexposure and diffusion. Typically under exposure occurs at the bottom of the sample. If under exposure is experienced, the developer will diffuse into this less dense region causing swelling and reduced stiffness. It can be best seen in relatively small features (figure continued).

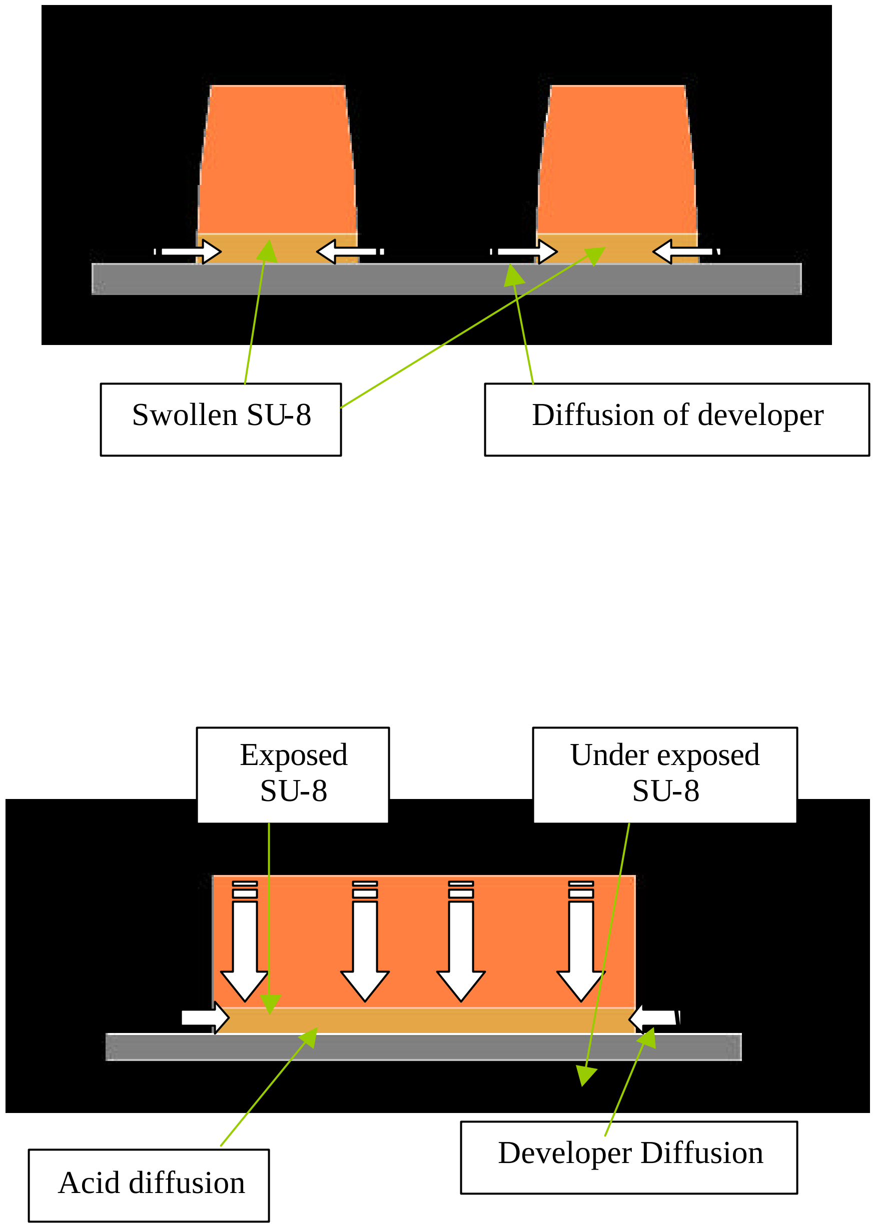
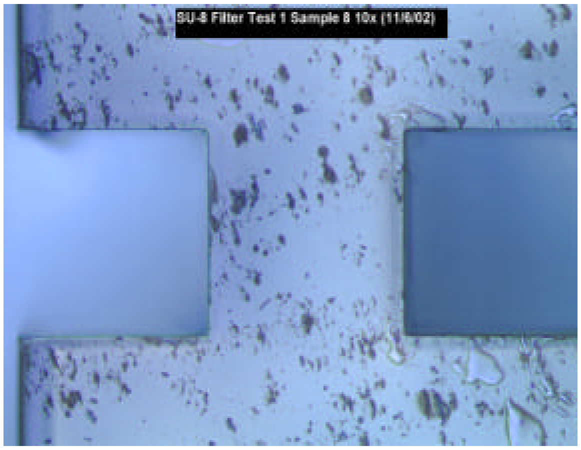


Figure 20: **Large exposure's resistance too underexposure. If the exposed SU-8 is large then the upper portion will diffuse acid into the lower part. Along with this and the reduced surface area to volume area of the underexposed region will enable the underexposed SU-8 to stay structurally sound.**

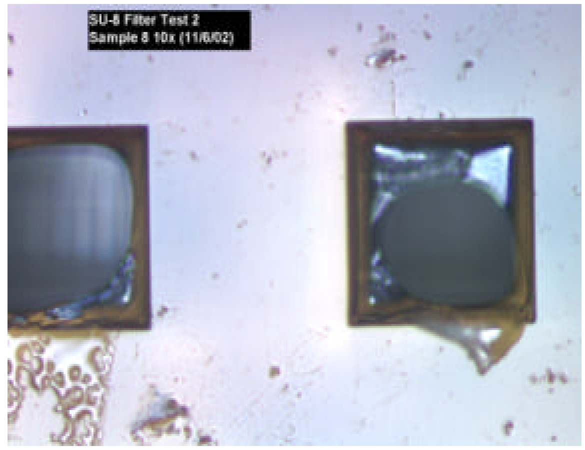
temperature increases (by the 2-3 order of magnitude near the glass transition temperature). However, if the post bake is not sufficient the SU-8 will not fully cross link and this will result in a weak structure. Post bake experiments were conducted for the single pour method as well as the chip casting method and both results are presented below. Initially, post bake experiments were conducted before the chip casting was developed. These experiments resulted in a post bake temperature that was much lower then the results of the chip casting tests. The reason for this was that the excessive solvent present in the single casted samples caused detrimental diffusion that could only be slowed by the use of a significantly lower post bake (50 °C). However, once the solvent content was properly reduced, using the chip casting method, the higher temperature (96 °C) produced the best results.

2.4.1 Post Exposure Bake for Single Pour Casting

All of the SU-8 samples presented in this section had a single pour casting which resulted in high uncontrolled solvent content. In one experiment, a sample was not post-baked while the other sample was post baked at the recommended temperatures of Microchem (96 ^{0}C). As shown in Figure 21, the sample that was not post-baked clearly gave a better result. Both of the SU-8 samples were 1.5 mm tall and exposed at CAMD's XRLM1 with an additional 63 μm of aluminum filtering (TBR 4.3) and a bottom dose of 30 J/cm^3 (the square holes are 475 μm wide and the sample was developed overnight). The sample that was not post-baked could have experienced a period at an elevated, but unquantified, temperature during the exposure itself since the scan length of the beam as short and the rate of energy absorption was greater than would normally be associated with an exposure with a longer scan length.



a)



b)

Figure 21: High Solvent Post Bake Results. Same exact procedure was done on two samples except one sample was baked at 96 °C (b) and the other was not baked at all (a). The non-baked test had much better results.

2.4.2 Post Exposure Bake Procedure for Single Pour Casting

Further tests were done to determine the equivalent post-bake temperature and time that would give the same results as the radiation heating. Doing exposures with the normal scan length were used for the experiments. The typical scan length is much larger than the one used in the experiment that involved no post bake described previously. A series of post-bakes were investigated, with the temperature being varied from room temperature to 50 °C, holding the exposure time constant at 20 minutes. CAMD preformed a similar study after this one was performed, and they came to the conclusion that 50°C for one hour gave the best results. The lower post bake temperature did allow for the higher solvent content samples to be more successful. The reduced temperature was necessary due to the increased diffusivity of acid in SU-8 when solvent content is high.

2.5 Exposure and Post Bake Test Matrix

This section discuss the post bake experiments that include the chip casting method which as discussed previously produce significantly enhanced structures when compared to the single pour method. Also in this section the proper dosage for exposures is also tested. These two processes were congruently tested due to their close interaction.

A matrix of 28 tests was performed consisting of all combinations of the three variables listed below, with the exception that experiments were not performed at 30 J/cm^3 for the 3 mm-thick SU-8 cast sheets.

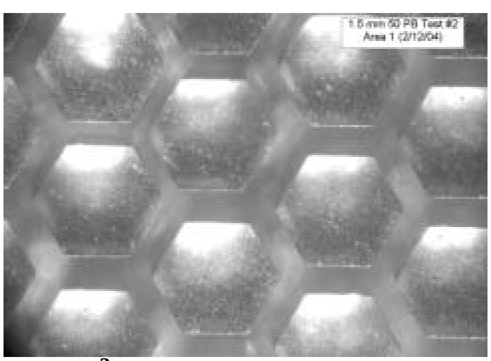
Bottom dose: 10 J/cm^3, 15 J/cm^3, 20 J/cm^3, 25 J/cm^3, 30 J/cm^3
SU-8 cast sheet thickness: 1.5 mm, 2 mm, 3 mm
Post bake temperature: 50 ^{0}C for 20 minutes, 96 ^{0}C for 20 minutes

In all the tests the TBR was equal to 4 (as the SU-8 sheet thickness was increased, the filtering was increased appropriately). Therefore, for a given bottom dose, the top dose was independent of sheet thickness. While the top dose was a function of only the bottom dose, the *duration* of the exposures was a function *both* of the bottom dose and the SU-8 thickness. For example, for a given bottom dose, the duration (assumed approximately proportional to the mA/min of the exposure) required to expose the 2.0 mm thick SU-8 sheet was a factor of 2.4 the duration required to expose the 1.5 mm thick sheet. The time required to expose the 3.0 mm thick SU-8 sheet was a factor of 7.5 greater than that required to expose the 1.5 mm thick sheet. In all tests, an x-ray mask was used that produced a densely packed array of SU-8 posts of hexagonal cross section with flat-to-flat dimension of 480 μm, and gap between adjacent posts of 125 μm.

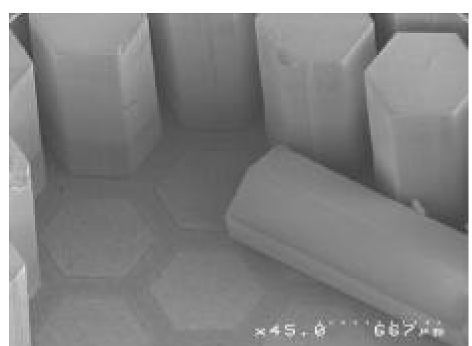
<u>1.5 mm tall features:</u>

Ten experiments were performed defining features with heights of 1500 μm. Five experiments utilized a 50 ^{0}C post bake, varying the bottom dose, while the other five experiments utilized a 96 ^{0}C post bake, also varying the bottom dose. For the 50 ^{0}C post bake experiments, the results were excellent at bottom doses of 10 J/cm^3, 15 J/cm^3, and 20 J/cm^3. However, at a bottom dose of 25 J/cm^3 there appeared to be the beginnings of significant residue between the posts and at 30 J/cm^3 there was significant undeveloped residue between the posts. Figure 22 shows the increasing presence of residue between the SU-8 features associated with increasing dose.

The same set of experiments was repeated with a post bake temperature of 96 ^{0}C. The results are shown in Figure 23. Again, the results are very good when the bottom dose was less than 20 J/cm^3. At higher doses, the result was still very good and much better than the case where the post bake temperature was only 50 ^{0}C. At higher dose, however, there was an increasing presence of a very thin film that covered the surface. The origin of this film is unknown, but it is not believed to be associated with lateral diffusion of cross-linking PAG into nominally unexposed regions. Overall, the higher post bake temperature produced less evidence of diffusion at the higher doses.

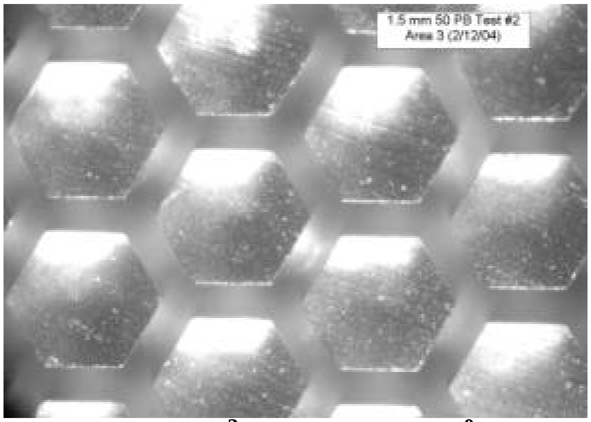


a) 10 J/cm^3, plan view showing no residue between posts

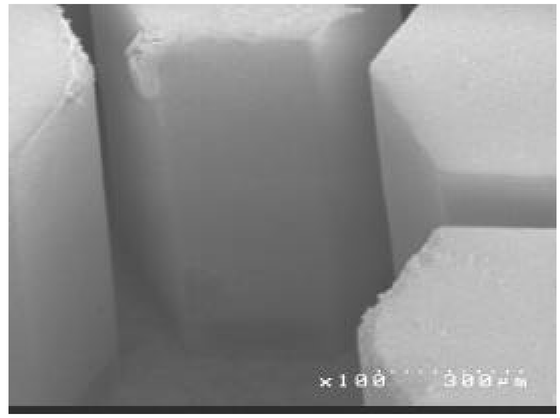


b) 10 J/cm^3, side view showing no residue between posts

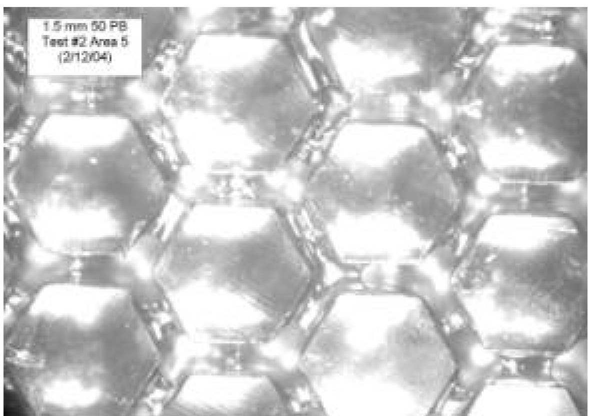
Figure 22: 1.5 mm tall features and post bake 50 ^{o}C, TBR =4 and varying bottom dose (figure continued)



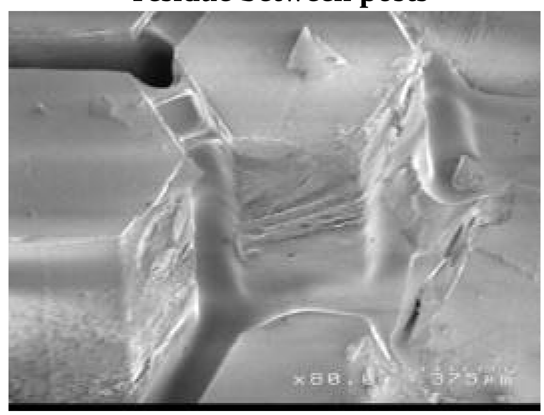
c) 20 J/cm^3, Post bake = 50 ^{0}C



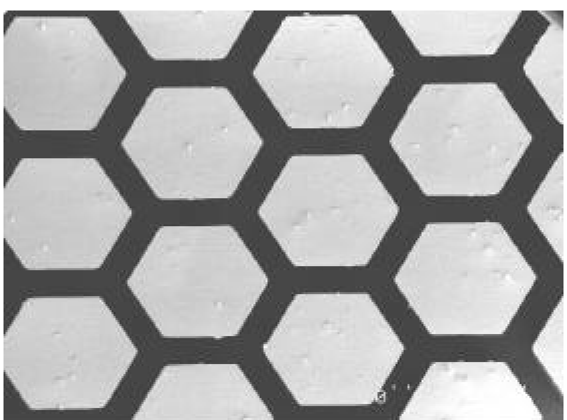
d) 20 J/cm^3, side view showing no residue between posts



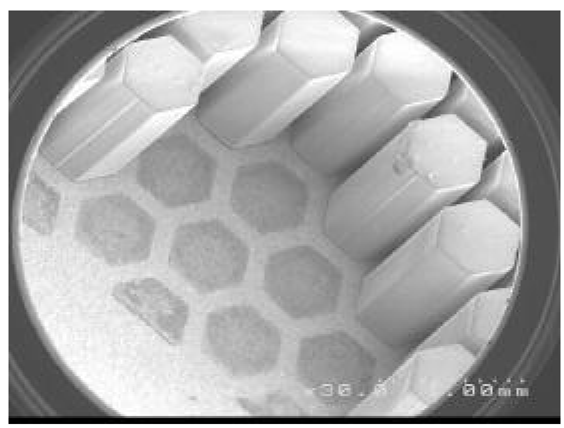
e) 30 J/cm^3, Post bake = 50 ^{0}C



f) 30 J/cm^3, side view significant residue between posts

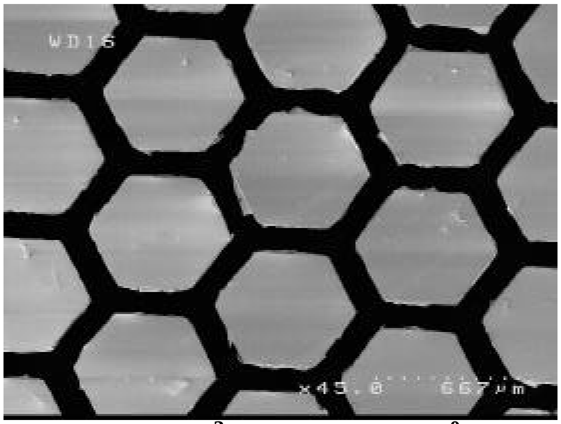


a) 10 J/cm^3, plan view showing no residue between posts

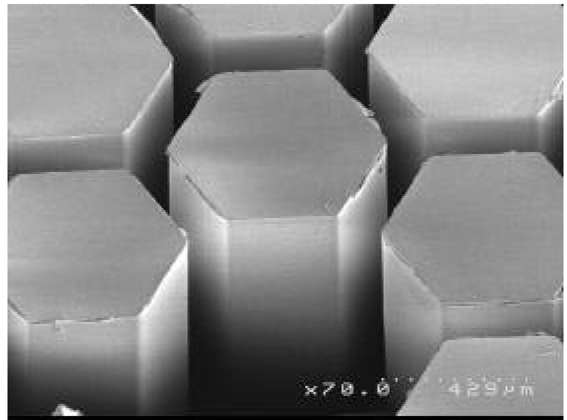


b) 10 J/cm^3, side view showing no residue between posts

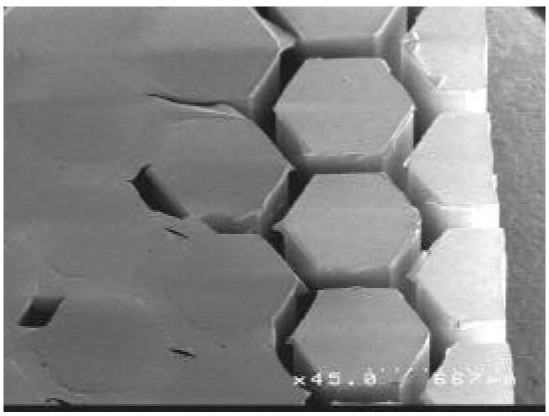
Figure 23: 1.5 mm tall features and post bake 96 ^{o}C, TBR =4 and varying bottom dose (figure continued)



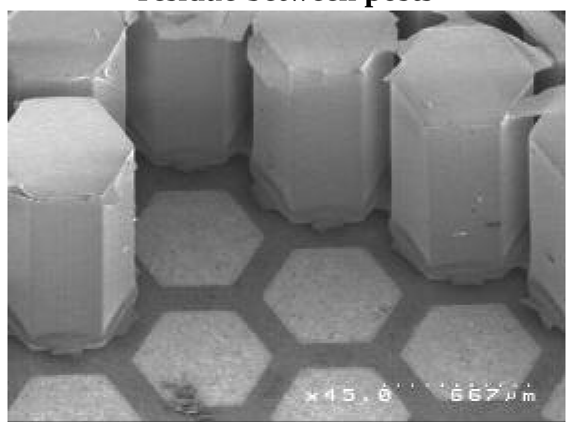
c) 20 J/cm^3, Post bake = 96 ^{0}C



d) 20 J/cm^3, side view showing no residue between posts



e) 30 J/cm^3, Post bake = 96 ^{0}C

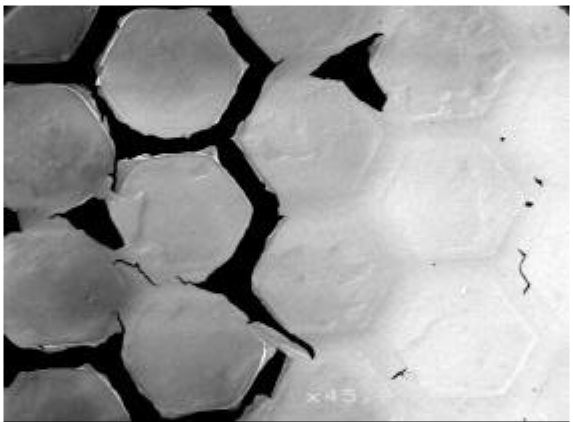


f) 30 J/cm^3, side view no residue and only a thin film between posts

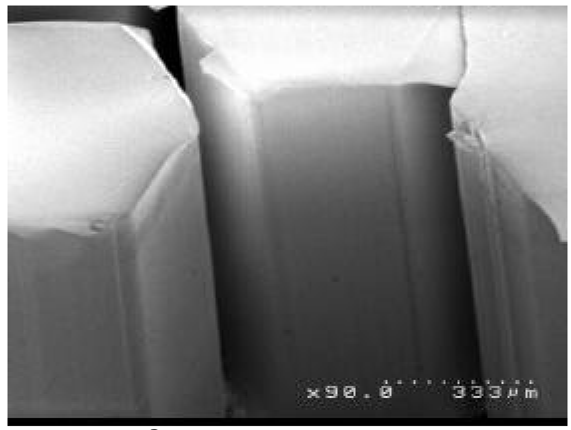
<u>2.0 mm tall features</u>:

Ten experiments were performed defining features with heights of 2000 µm. Five experiments utilized a 50 ^{0}C post bake, varying the bottom dose, while the other five experiments utilized a 96 ^{0}C post bake, also varying the bottom dose. The results are shown in Figure 24. For the 50 ^{0}C post bake experiments, a thin film connected the tops of the SU-8 features. Beneath the film, the development was complete. At a 20 J/cm^3 bottom dose, significant undeveloped SU-8 remained between the posts, and the problem worsened when the bottom dose was increased to 30 J/ cm^3.

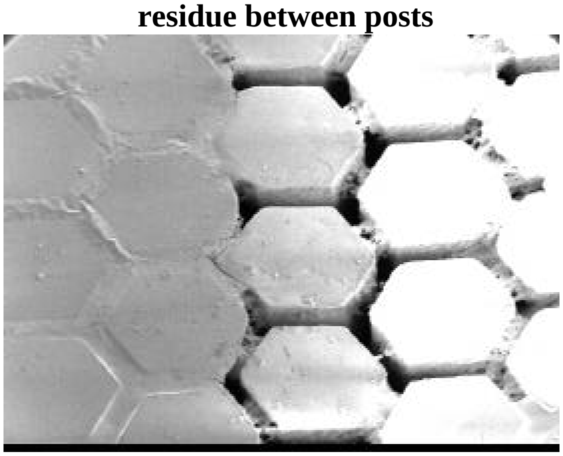
As was the case with the 1500 μm tall features, the experiments that utilized a post bake temperature of 96 ^{0}C produced better results. The results are shown in Figure 25. At all doses, a thin film between the posts was noticed at the surface. The thickness of this film was relatively uniform, but it increased slightly with increasing bottom dose. Beneath the film, at low doses, the SU-8 was removed completely. As the dose was increased, there was increasing, sometimes substantial, volumes of SU-8 remaining between the posts.



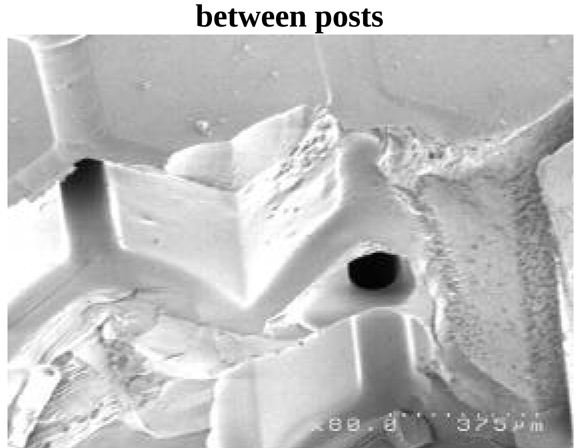
a) 10 J/cm^3, plan view showing no residue between posts



b) 10 J/cm^3, side view showing no residue between posts

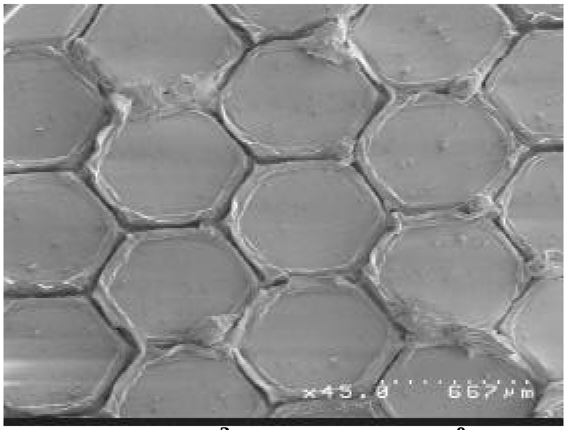


c) 20 J/cm^3, Post bake = 50 ^{0}C

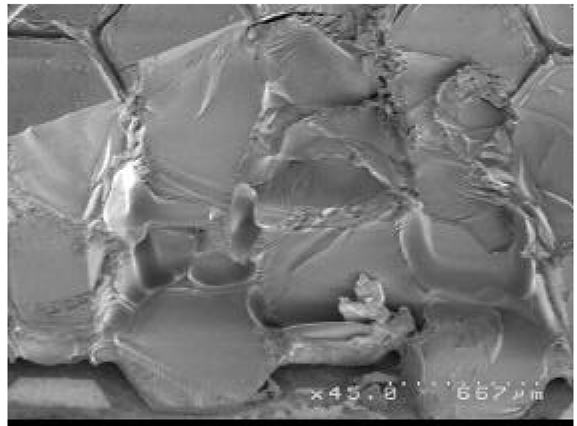


d) 20 J/cm^3, side view showing residue between posts

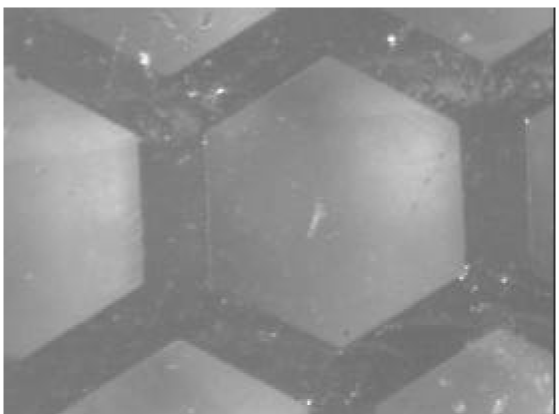
Figure 24: 2.0 mm tall features and post bake 50 ^{o}C, TBR =4 and varying bottom dose (figure continued)



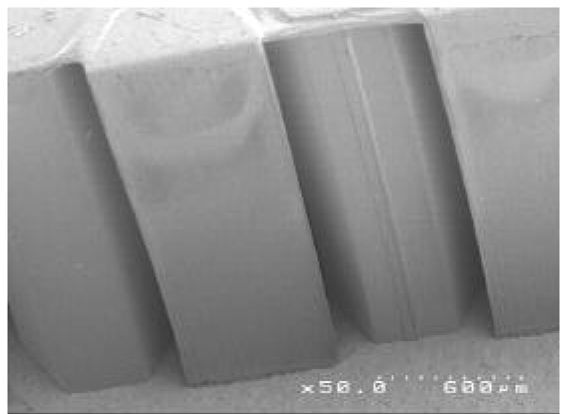
e) 30 J/cm^3, Post bake = 50 ^{0}C



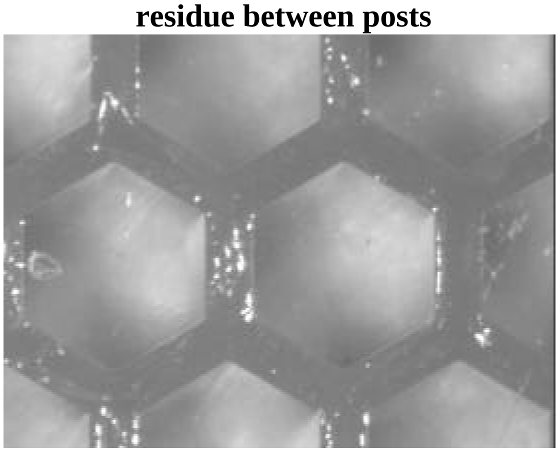
f) 30 J/cm^3, side view significant residue between posts



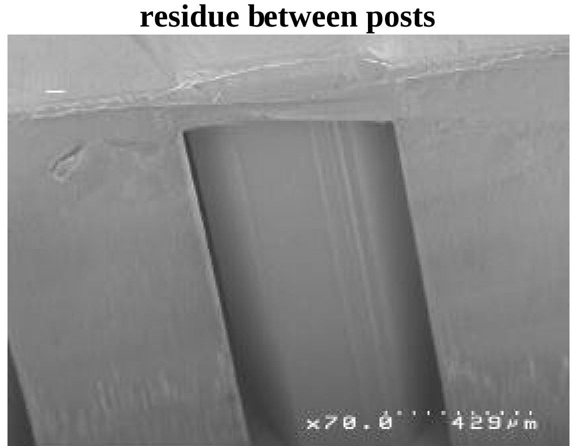
a) 10 J/cm^3, plan view showing no residue between posts



b) 10 J/cm^3, side view showing no residue between posts

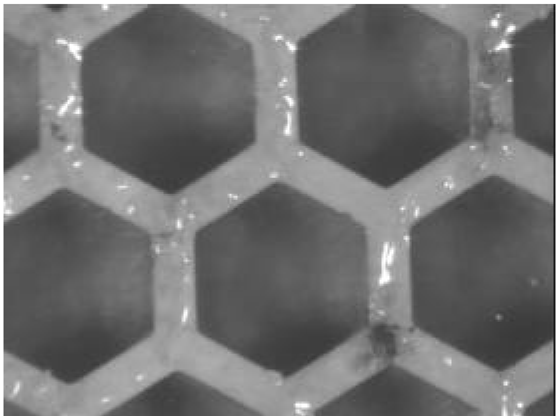


c) 20 J/cm^3, Post bake = 96 ^{0}C

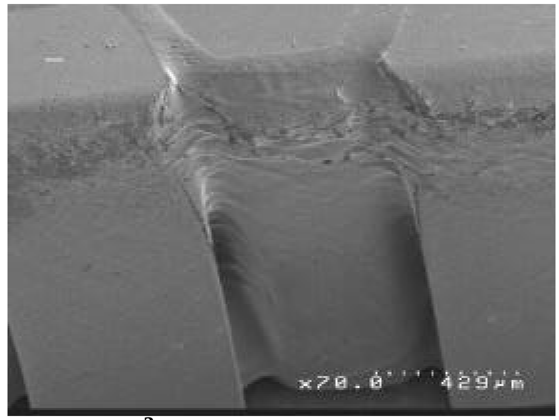


d) 20 J/cm^3, side view showing no residue between posts

Figure 25: 2.0 mm tall features and post bake 96 ^{0}C, TBR =4 and varying bottom dose (figure continued)



e) 30 J/cm^3, Post bake = 96 ^{0}C



f) 30 J/cm^3, side view significant residue between posts

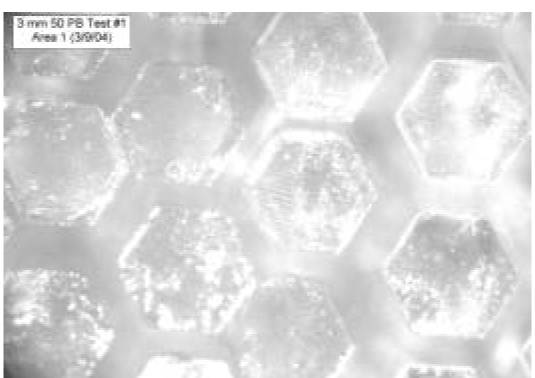
<u>3.0 mm tall features</u>:

Four experiments (bottom dose 10 J/cm^3, 15 J/cm^3, 20 J/cm^3, and 25 J/cm^3) were performed using a post bake temperature of 50 ^{0}C. The results were not good. Over the entire range of bottom dose tested, extensive undeveloped SU-8 existed between the posts, even at the lowest bottom dose value of 10 J/cm^3. Also, as shown in Figure 26, the post spacing was not uniform, indicating that the posts were bending.

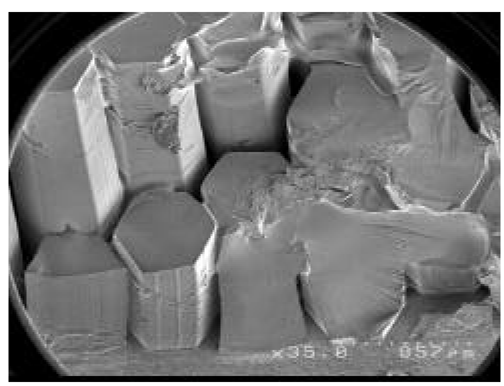
An identical set of four experiments were performed, but using the traditional post bake temperature of 96 ^{0}C. At a bottom dose of 10 J/cm^3, the results were quite good, with only a light film between the tops of the posts noticed in certain regions (Figure 27). As the bottom dose was increased, the definition of features remained excellent, but the thickness of the region of undeveloped SU-8 between the tops of the posts becomes thicker. Beneath this film, over the entire range of dose tested, the SU-8 was developed properly.

Overall, the experiments at lower dose using the 96 ^{0}C post bake were very successful, with the main problem being the thin surface film between the SU-8 features. This thin film seems to have a different origin than much thicker layers of undeveloped

SU-8 that characterizes some experiments. To differentiate between the thin film phenomenon and the more substantial thicker undeveloped SU-8 residue that is sometimes noticed (and believed to be related to PAG diffusion), an additional set the experiments was performed. In this set of experiments, the height of the SU-8 casting was increased slightly to 3.25 mm. After the exposures/post bake procedure was completed, the top 250 micrometers of the SU-8 casting were removed (via fly cutting), leaving the desired SU-8 feature height of 3.0 mm. It should be noted that the chip casting procedure produces an SU-8 casting that is hard and easily machinable. Prior to incorporating this procedure, it was difficult to machine the SU-8, especially when the thickness of the SU-8 casting exceeded 2 mm. As can be seen in Figure 28, excellent results were achieved when the bottom dose was 10 or 15 J/cm^3. The fly cutting procedure removed the thin film that is often present, leaving well developed, very high aspect ratio features (3 mm tall, the gap between posts equal to 125 μm). At a bottom dose of 25 J/cm^3, there was substantial undeveloped SU-8 remaining within a layer that was hundreds of micrometers thick.

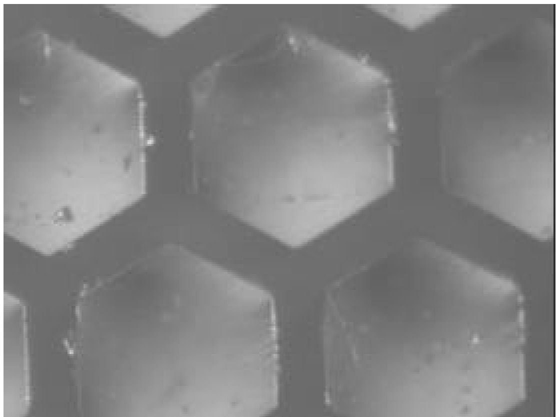


a) 10 J/cm^3, plan view showing residue between posts and bending of posts

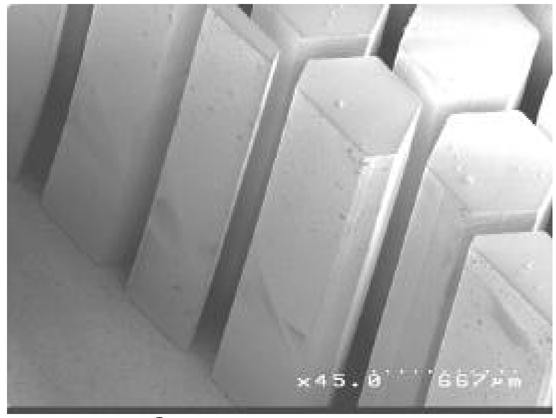


b) 20 J/cm^3, plan view showing more extensive residue between posts and bending of posts

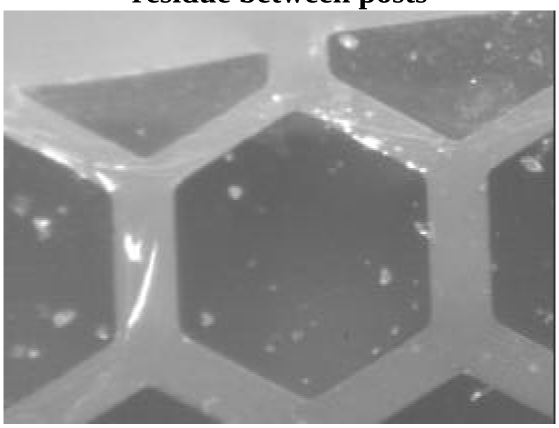
Figure 26: 3.0 mm tall features and post bake 50 ^{o}C, TBR =4 and varying bottom dose



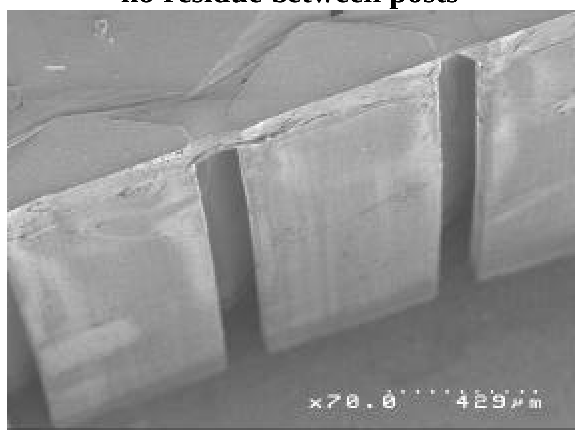
a) 10 J/cm^3, plan view showing no residue between posts



b) 10 J/cm^3, side view near edge showing no residue between posts

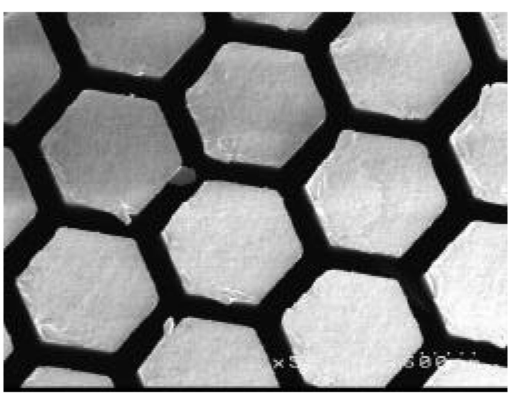


c) 20 J/cm^3, Post bake = 96 ^{0}C-thin film connecting tops of features

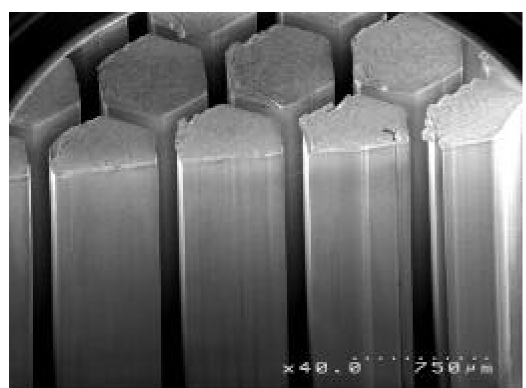


d) 25 J/cm^3, side view showing thicker cap and increased residue below cap between posts

Figure 27: 3.0 mm tall features and post bake 96 ^{o}C, TBR =4 and varying bottom dose

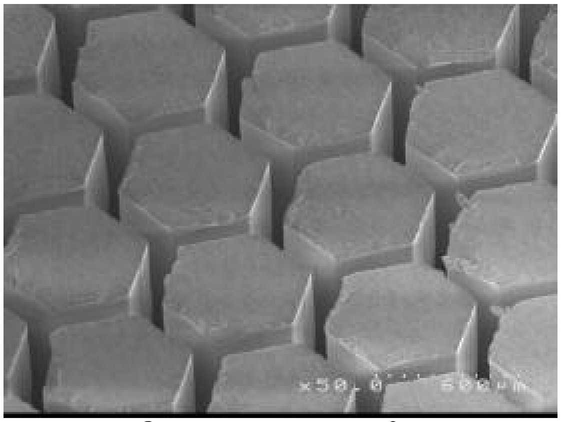


a) 10 J/cm^3, Post bake = 96 ^{0}C, top 250 μm fly cut

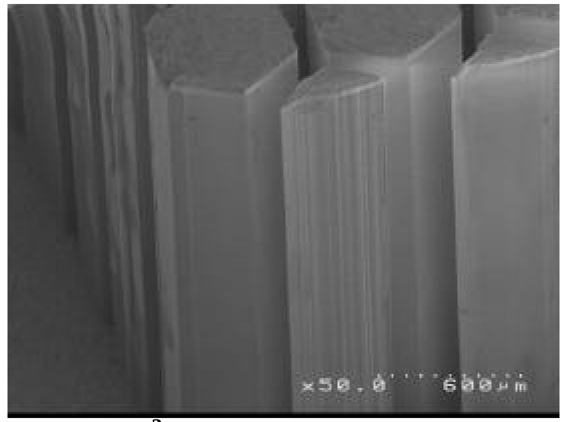


b) 10 J/cm^3, side view showing no residue between posts

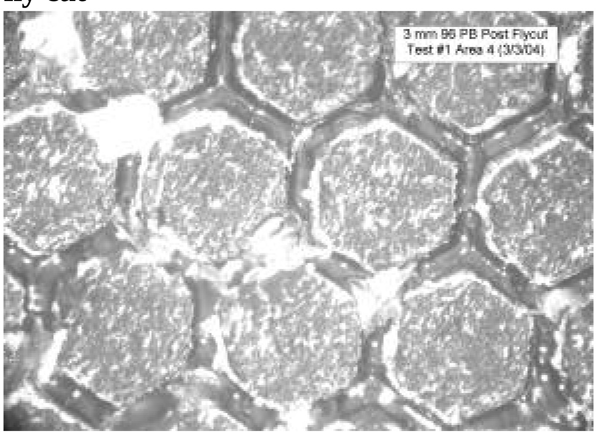
Figure 28: 3.0 mm tall features after post bake 96 ^{o}C, and subsequent fly cut to remove top 250 μm. (TBR =4). (figure continued)



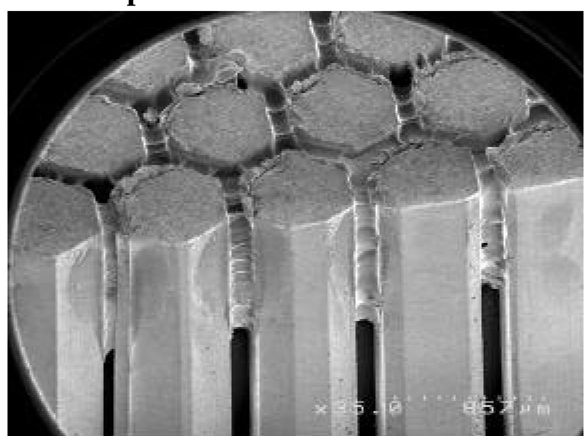
c) 15 J/cm^3, Post bake = 96 ^{0}C, top 250 μm fly cut



d) 15 J/cm^3, side view showing no residue between posts



e) 25 J/cm^3, Post bake = 96 ^{0}C, top 250 μm fly cut



f) 25 J/cm^3, side view showing considerable undeveloped SU-8 between posts

2.5.1 Exposure and Post Bake Test Matrix Conclusions

When the solvent content is 7% and uniform, the post bake temperature of 96 ^{0}C produces better results than the 50 ^{0}C post bake temperature. This result differs from that noted in the single casting post bake section, when solvent content was higher and less well controlled, and when the height of the SU-8 features was shorter (i.e. 1.5 mm). A plausible explanation is that when the solvent content is reduced, the effective diffusivity of PAG through the exposed SU-8 is much lower. As a result, the advantages associated with increased cross linking at 96 ^{0}C outweigh the fact that diffusivity through the resist nominally increases with temperature. At higher solvent content, the reverse may be true, explaining why some experiments showed better results when the post bake temperature was 50 ^{0}C. Achieving a uniform, low solvent content is a very important starting point to successfully define tall, dense, high aspect ratio SU-8 features. Future tests need to be performed to investigate whether solvent content at even lower values than 7% produce even better results.

For the geometry studied (maximum height = 3000 μm, gap = 125 μm) substantial undeveloped SU-8 appeared between the posts at top doses around 90-120 J/cm^3 (top dose equals the product of bottom dose and TBR). The thick undeveloped deposits of SU-8 between posts is attributed to diffusion. For the feature pattern analyzed, the top dose needed to remain below 50 J/cm^3 to eliminate the effects of diffusion. It should be noted that this value depends upon the geometry of the features. For the case of sparsely spaced features, the adverse effects of diffusion will not be noticed unless the top dose is much higher, while much smaller gaps may require an even smaller top dose.

The typical exposure time for a similar 3 mm sample discussed previously at CAMD's XRLM 2 for a centimeter of exposure would be 40 minutes (5026 mA.min/cm). When doing a large exposure area would translate into a very long required exposure time. However if a "wiggler" like XRLM 4 at CAMD is used the time significantly drops. For CAMD's XRLM 4 running at 7 Tesla the same exact exposure would only take 6 seconds (12.7 mA.min/cm). Below in Figure 21 the pictures of the above experiment are presented.

An undesired SU-8 film sometimes appears at the surface of the features that is not attributed to diffusion. The presence of the film was a much stronger function of *the duration of the exposure* than any other variable that could be identified. The cause of the film is not known, but some possible theories that are listed below can be ruled out:

1. Leakage through the mask: The mask absorber patterns used in these experiments were gold and 80 μm thick. The contrast of the mask was very high, very little radiation leaked through the gold, and the spectrum that did leak through is very hard and would have exposed uniformly the volume of SU-8 shaded by the absorber pattern. There is no known explanation why leakage would cause the thin film phenomenon.

2. Allowing the SU-8 to be exposed to unfiltered light with high absorption: If the SU-8 were exposed to unfiltered light (perhaps some unknown source of unfiltered light exposed the upper surface of the SU-8 uniformly), then PAG might be generated within a thin film over the entire SU-8 cast film surface. However, 4 or 5 exposures were performed on different regions of each SU-8 cast sample, with the only the bottom dose varying. After the exposures were completed, the sample was post baked and developed. Great discrepancies in the prevalence of the surface film were noted on the same sample, with the film always more prevalent when the dose (and time of exposure) was greater. An unknown source of light would be expected to induce to the same degree the unwanted film in all the exposures of a single casting.

Presently, the prevailing hypothesis to explain the film formation is that a reactive gaseous species is produced during exposure above a very localized surface of the SU-8. This reactive species locally produces PAG that subsequently initiates cross linking at the surface. This explanation would be consistent with the observation that the film is more prevalent as the duration of the exposure is increased. It is also consistent with the observation that the film is not seen when the high energy “wiggler” beamline at CAMD is used (where the times of exposure are very short relative to those discussed in paper).

Until the cause of the thin film is diagnosed, a post machining process can be used to greatly minimize its adverse effects. The post machining process also offers the advantage that the heights of the SU-8 features can be very accurately controlled since whatever vertical shrinkage that is often associated with the post bake is no longer an issue. Again though, the machining process is only feasible if the solvent content issue has been properly addressed.

2.6 Development

If all of the previous steps are properly completed to minimize PAG diffusion, the development should be simple for most exposures. For these cases the sample should be suspended face down over spin bar with a slow spin rate for 3-6 hours. Otherwise, when the sample is a difficult pattern/height combination and/or the other steps in the processing were not optimized the following can be done during the development procedure to improve the results:

- **Problem:** Swelling, softening, and/or bending of exposed SU-8
    - **Procedure:** Decrease the agitation rate and the duration of the development. The sample should be carefully monitored and once all of the unexposed SU-8 is developed the sample should be immediately removed and dried. This will decrease developer diffusion into the under cross-linked SU-8. The lower agitation rates will prevent some of the bending of the exposed SU-8 features.

- **Problem:** Undevelopable SU-8 in unexposed regions
    - **Procedure:** Increase the development time and the agitation speed. This will allow for mechanical assistance in the removal of the undevlopable SU-8.

3. Final Results

The main premise of this thesis was that the diffusion of acid into unexposed regions prior to and during post bake is THE important physical parameter that governs all SU-8 processing steps. The verification of this premise has been exemplified by the previously discussed theoretical models and conducted experiments. A simply constructed model initially proved the plausibility of diffusion. As stated earlier, the diffusivity of a resist can vary by three orders of magnitude in a 20 °C range, when the glass transition temperature is included in the temperature range. Since the typical post bake recommended by the company that manufactures SU-8 is using a temperature of 95 °C (resulting in over a 70 °C temperature change), under typical processing procedures a sample can experience a dramatic increase in temperature. Heating to elevated temperatures will increase the diffusivity dramatically. This can be seen in Figure 2 where the increase in diffusivity of four orders of magnitudes has detrimental affects. This diffusion causes immense amounts of PAG to migrate into the unexposed regions making them undevelopable. From the model it can be ascertained that if diffusion is minimized the acid will not diffuse into the unexposed areas. The other source of increasing and decreasing diffusivity can come from the material property. In SU-8 this can be greatly altered by the solvent content.

The affects of diffusion that the model theoretically predicted were seen in the SU-8 exposure experiments that were accomplished. After the realization that diffusion of PAG was the mechanism that was limiting the patterns/height combinations that were being tried, experiments were conducted to discover how to minimize diffusion in all of the SU-8 processing steps. The following is a list of all SU-8 processing steps along with the corresponding diffusion mechanism and resulting procedure that diminishes PAG diffusion.

3.1 Pre Bake

Diffusion Consideration: Solvent content is the largest physical parameter that must be controlled in order to decrease diffusion. Excess solvent in the SU-8 could allow for a less resistant path for the acid to diffuse through and/or it could also change the material properties of the SU-8 and increases diffusion. Although it is not known for certain how solvent affects diffusivity of PAG, it is known through experiments that excess solvent content does increase diffusion of PAG into the unexposed regions. Consequently, this results in undevelopable SU-8 where no SU-8 should remain.

Diffusion Limiting Procedure: The solvent content could not be minimized in a single cast so an alternate procedure must be used. A single thin cast will be done in order to reduce the SU-8 to a low uniform solvent content. After the thin cast has been accomplished, the SU-8 is mechanically removed from the surface of the casting apparatus. The SU-8 "chips" are put in another casting jig that forms the SU-8 "chips" into the desired shape. Simultaneously vacuuming and heating the SU-8 forms the desired shape. This procedure not only gives a precise solvent content it also results in a very uniform solvent profile throughout the SU-8 sample.

3.2 Exposure

Diffusion Consideration: The two ways that diffusion can be experienced during exposures is by over exposing and under exposure. In over exposed SU-8, large amounts of acids will be created in the exposed area and this will create a large concentration gradient between the exposed and unexposed SU-8. PAG will then more readily diffuse into the unexposed area and result in undevelopable SU-8 where no SU-8 should remain. Underexposure is problematic because during development the developer will more readily diffuse into the under cross-linked SU-8. When SU-8 absorbs developer this makes SU-8 soft and structurally weak.

Diffusion Limiting Procedure: Previously thick SU-8 samples had a high TBR, which created a wide range of doses in a single sample. Thus it was difficult to determine the optimal dose for SU-8. From a series of experiments it was determined that the proper bottom dose is 10 J/cm^3 and upper limit of allowable top dose is 50 J/cm^3 for the given dense pattern.

3.3 Post Bake

Diffusion Consideration: During post bake the elevated temperature for a time period can greatly increase diffusivity. It has been previously mentioned, that the diffusion rates in a resist can increase by three orders of magnitude in a 20 °C range when the glass transition temperature is included in the range. Before the solvent content was reduced to low/uniform levels lower temperatures had to be employed in order to control diffusion. However after the solvent reduction casting was implemented the lower temperature's faults were seen.

Diffusion Limiting Procedure: Through testing it was found that the best post bake procedure was to start at 60 °C then ramp the temperature to 96 °C and hold the temperature for twenty minutes. After the sample is sufficiently cooled to room temperature, the sample is then fly cut by approximately 200 µm to remove the thin film that was discovered.

3.4 Development

Diffusion Consideration: Development is only indirectly related to diffusion. If all of the other steps in the process were completed to minimize diffusion, the development is not that critical. However, if these steps were not properly optimized to limit diffusion or very difficult pattern/height combinations are being attempted, the development process is very important. Strong agitation and prolonged development time can help remove small amounts of undesired undeveloped SU-8. On the other hand, strong agitation and extra development time can harm the sample if the structures are underexposed and/or the structures are frail.

Diffusion Limiting Procedure: The general rule is that a sample should be subjected to high agitation forces and extra time of development if the geometry of the structures are holes in SU-8 and/or robust posts. Typically when small posts are desired it is difficult to submitted the sample to severe agitation and a prolonged time in the developer because the SU-8 post have a high tendency to fall off the substrate and deform in shape.

The geometry that was used during the majority of these tests was a pattern with a high density of posts. This type of geometry is one of the most difficult due to short diffusion lengths and the structural instability of high aspect ratio posts. The new alterations in the SU-8 processing steps, in order to limit diffusion of PAG, have allowed for the completion of previously unobtainable SU-8 structures. In order to gain some perspective on these new accomplishments, previous results will be shown here. Figure 29 below shows some of the previous accomplishments at LSU using SU-8. These posts are 1.5 mm tall, but more importantly these posts are not densely packed. The limited thickness of the SU-8 and the low density of the post diminished diffusion effects that can be seen in thicker denser SU-8 patterns. This is a typical example of prior results in which the patterns had low density of posts and exposures were short in height.

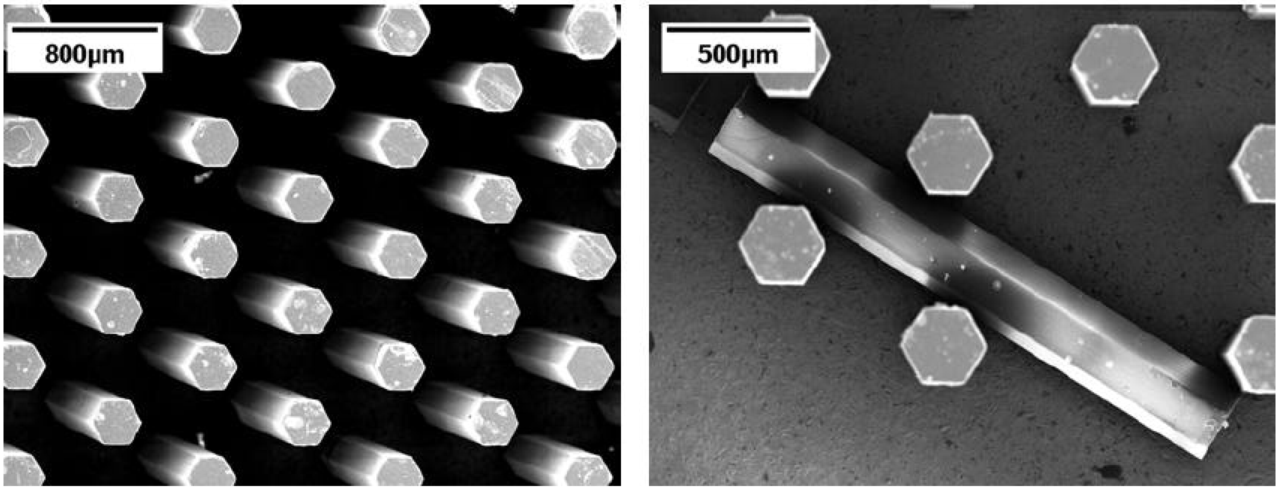
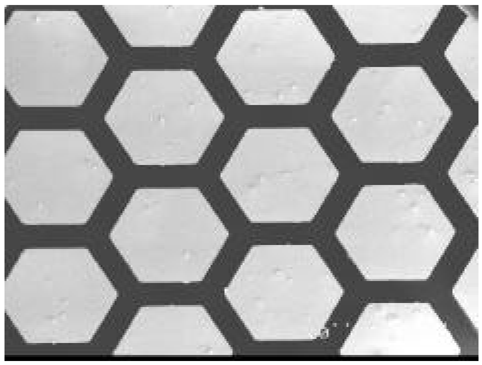
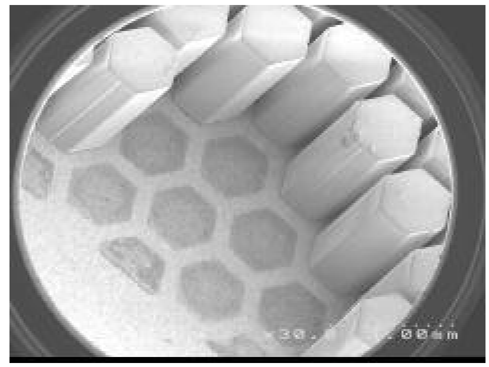


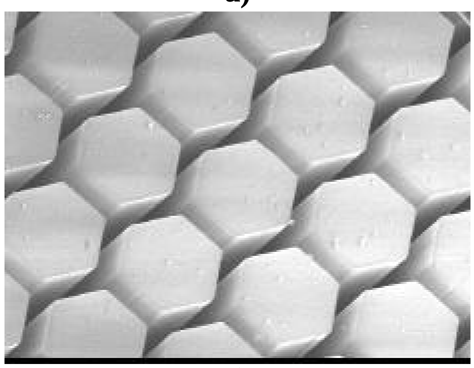
Figure 29: Previous results accomplished before diffusion limiting procedure was used [15].

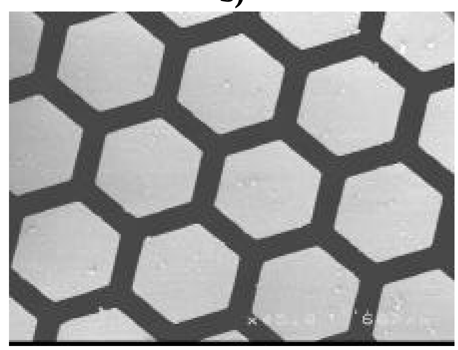
The results from using the diffusion limiting exposures will be placed in two groups. The first group is a collection of completed SU-8 exposures, which resulted in a dense field of posts. The posts are hexagons with diameters of 480 μm and the spacing between the posts is 125 μm. Three samples are included in this section that all had multiple exposures done on a single SU-8 sample. The first being a chip cast sample of only 1.5 mm in Figure 30 which was exposed on CAMD's XRLM1 with a TBR of 4.19. The bottom dose used was 10 J/cm^3 and the post baked using the 96 °C procedure. Afterwards the 1.5 mm was developed for 3 hours. The second is a sample of 2 mm shown in Figure 31. The sample was also exposed on XRLM1 with the same casting, TBR, post bake, development, and bottom dose as the 1.5 mm sample. The final sample is a 3 mm sample (Figure 32). The last sample is not a single exposure. This is the same sample that was previously discussed in the exposure section. As described before the sample had multi exposures to separate areas in order to determine proper exposure parameters.





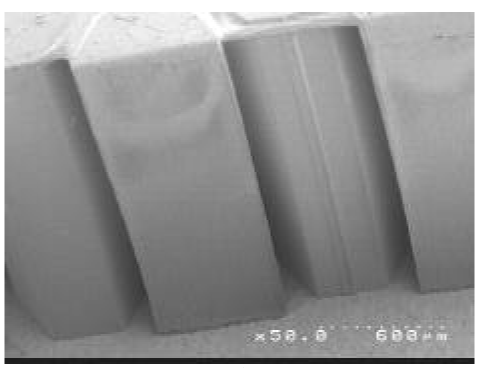
a) **b)**

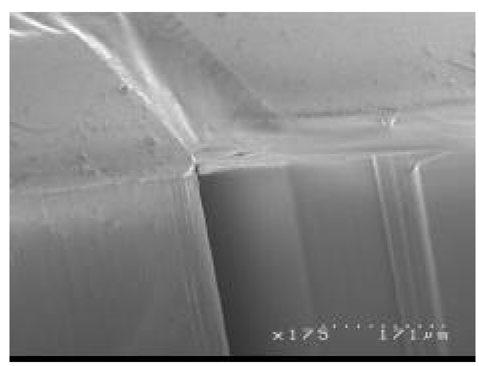




c) **d)**

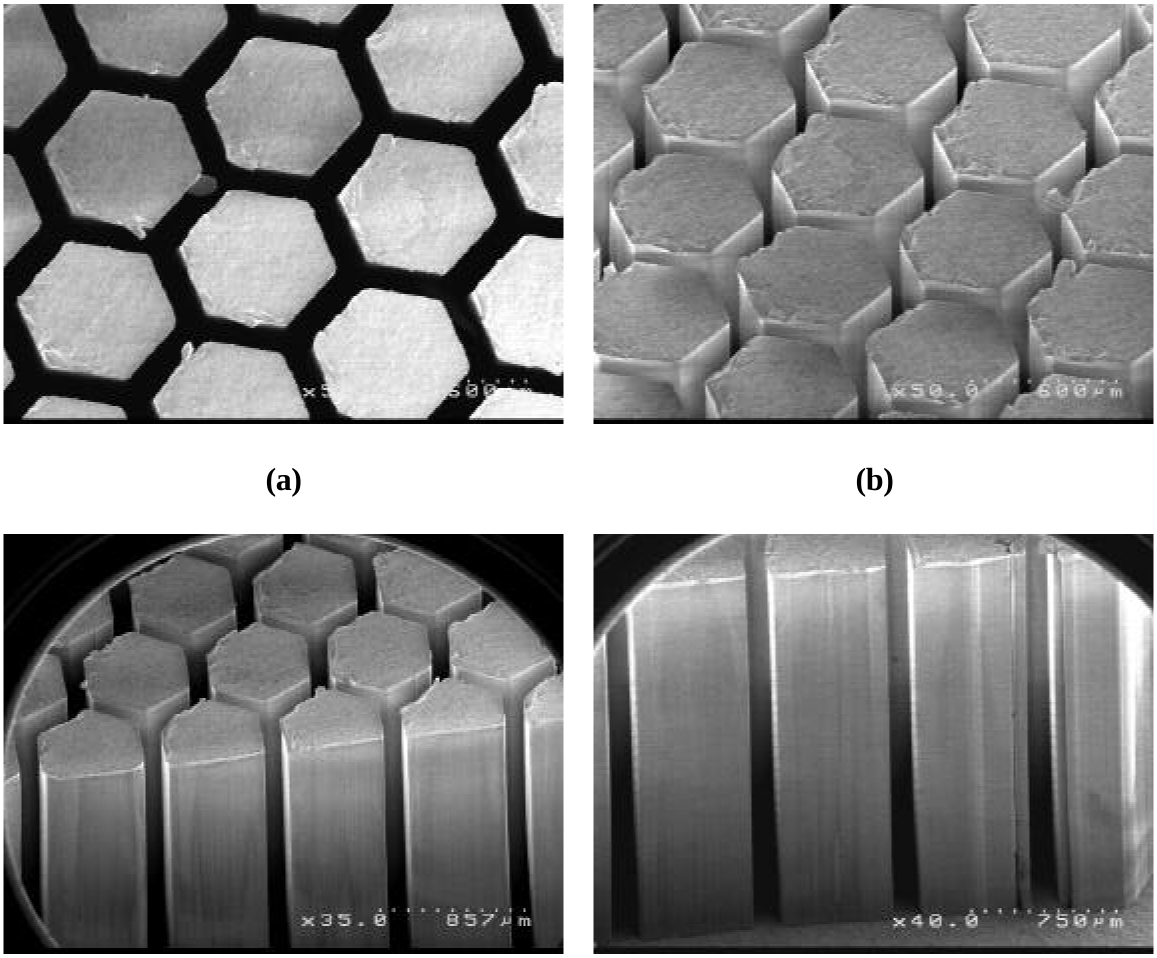
Figure 30: 1.5 mm tall sample with a bottom dose of 10 J/cm^3 and TBR of 4.





a) **b)**

Figure 31: 2 mm tall field of posts with a bottom dose 10 J/cm^3 (This sample has a thin film on the upper surface that can be easily remove with post exposure fly cutting).

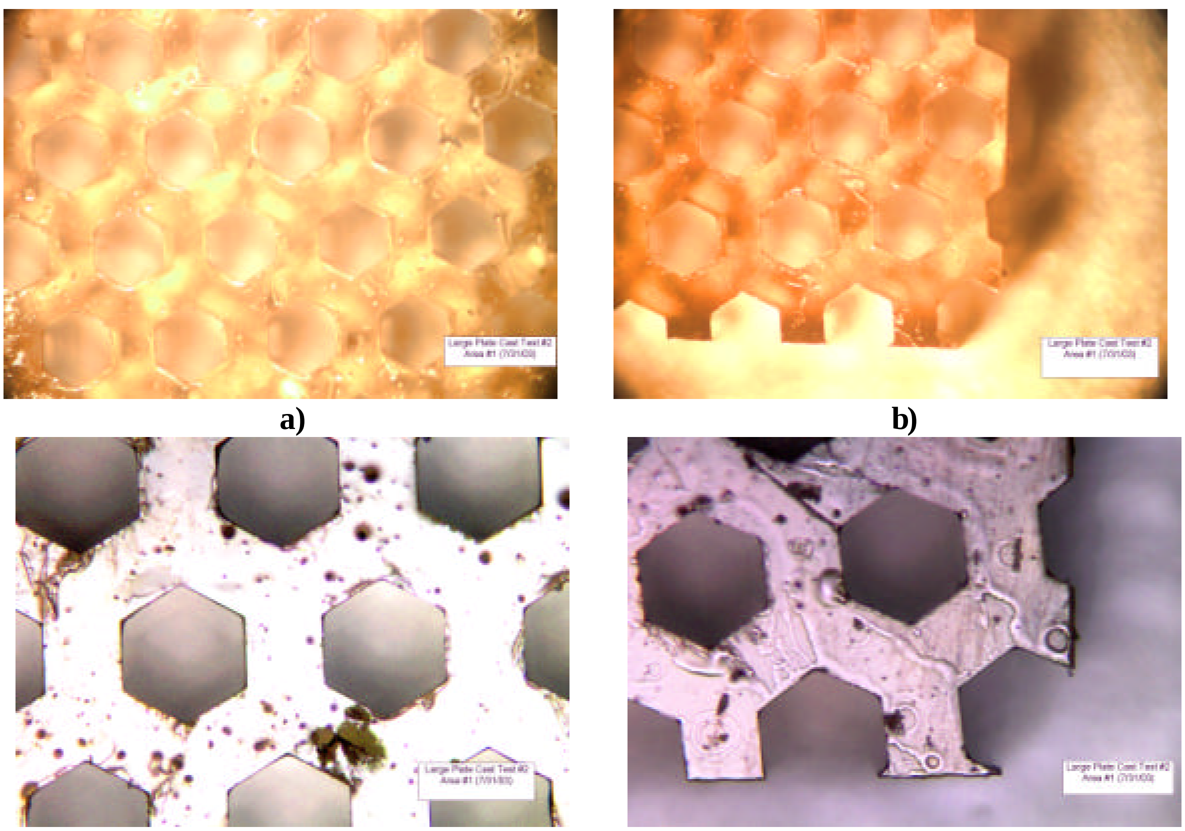


(a) **(b)**

(c) **(d)**

Figure 32: 3 mm SU-8 posts with a bottom dose of 10 J/cm^3 and a TBR of 4. This sample was post exposure fly cut from 3.25 mm down to 3 mm in order to remove the thin film that remained.

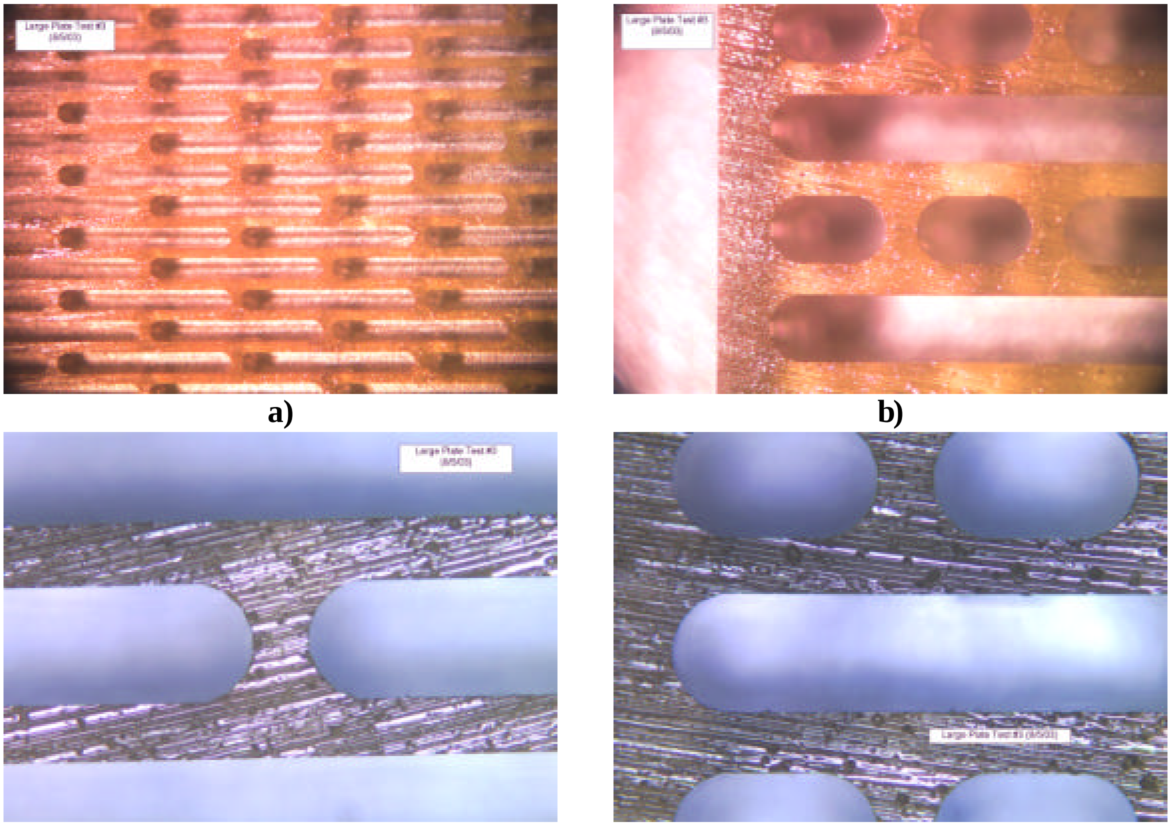
The second group of exposures is from experiments done using the diffusion limiting procedures on SU-8 exposures with geometries other then the main pattern used of tightly packed posts. These patterns were used to demonstrate the ability to successfully accomplish alternate patterns. Figure 33 below is photographs from a 2.6 mm exposure of 500 µm hexagon holes spaced 420 µm apart. The bottom dose was 10 J/cm^3 and the TBR was 8.17. The next set of photographs in Figure 34 show slots with a depth of 2.5 mm, wall thickness of 300 μm, channel width of 700 μm, and channel length of 5.5 mm. This sample was exposed with a bottom dose of 10 J/cm^3 and a TBR of 11. The final pattern that was done using the new procedure was done using a test pattern that had a wide variety of geometries and sizes. Using this pattern Daniel Berhardt accomplished six exposures on a single sample. SU-8 samples were exposed at heights of 4.5 mm (Figure 35, 36, and 37), 4 mm , and 2.5 mm. For each of the samples three pairs of exposures were done with varying bottom doses of 10 J/cm^3, 20 J/cm^3, and 30 J/cm^3 (the TBR was range between 3-5). Throughout his exposures the TBR was kept between 3 and 5. As one looks at the exposures it can be seen that as the bottom dose increases so does the diffusion of the PAG. Also the lower bottom doses experience more deflections. Following the previously chapters conclusions.



a) b)

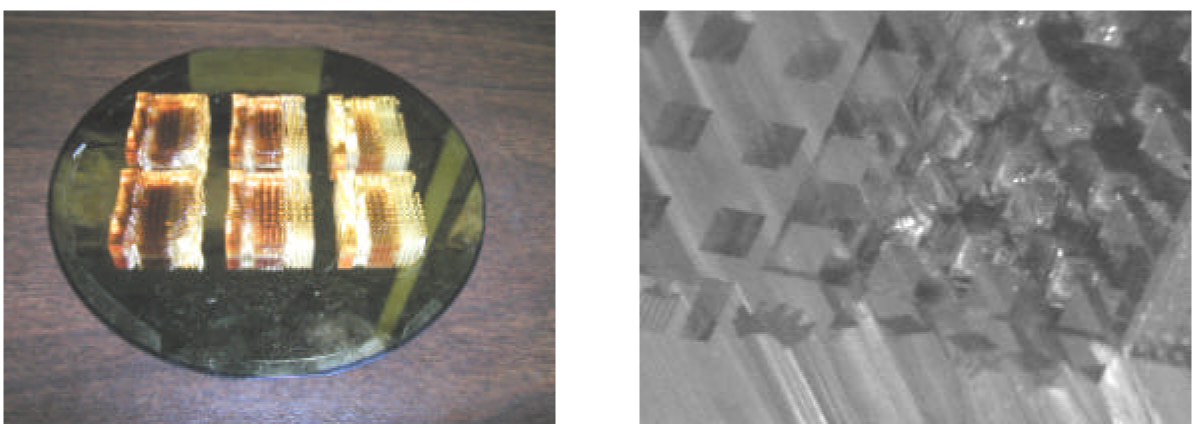
c) d)

Figure 33: 2.6 mm sample with 500 μm holes spaced 420 μm apart.



a) b) c) d)

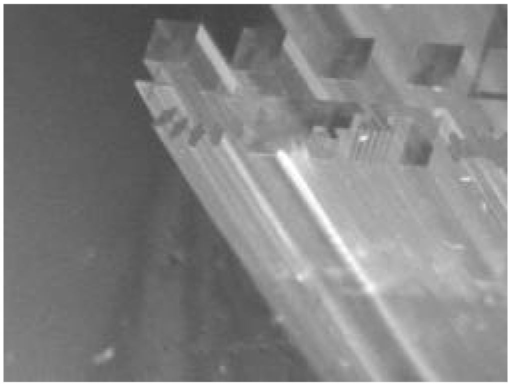
Figure 34: 2.5 mm tall channels in SU-8 with wall thickness of 300 μm, channel width of 700 μm, and channel length of 5.5 mm.

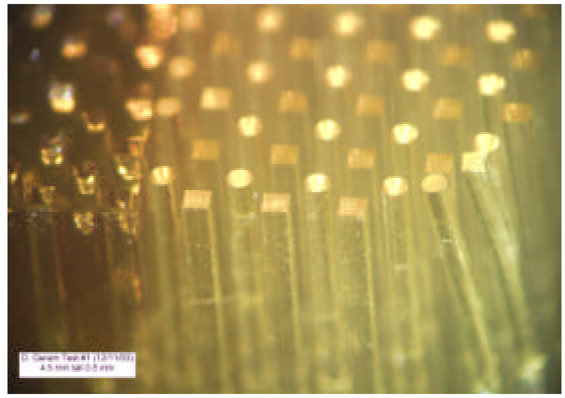


a) Overview of Exposure

b) Outer square 800 μm holes open and inner holes closed due to higher PAG concentrations near inner holes

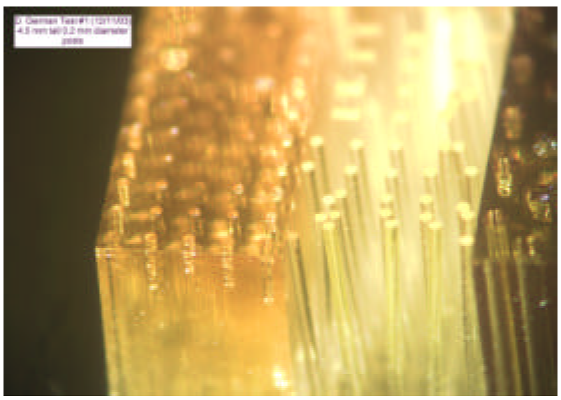
Figure 35: 4.5 mm sample with a bottom dose of 10 J/cm^3. (figure continued)



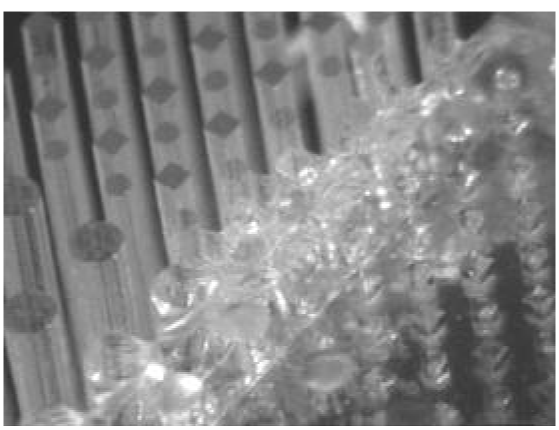


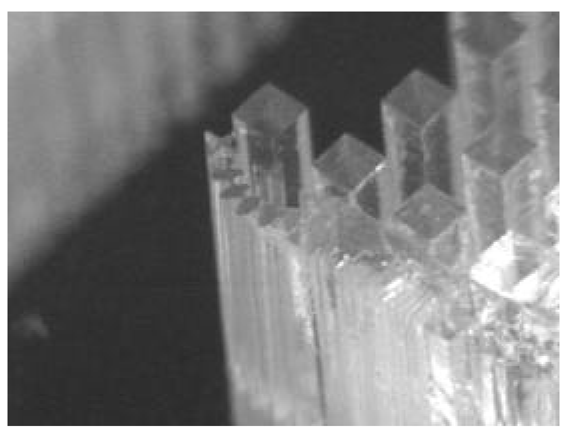
c) Three crosses from left to right 60 μm 50 μm and 40μm

d) 400 μm square posts. Posts on outer edge are bent



c) 200 μm diameter post that are more significantly bent the posts in b

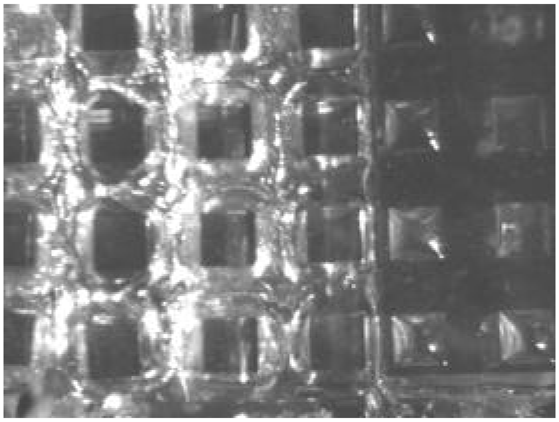




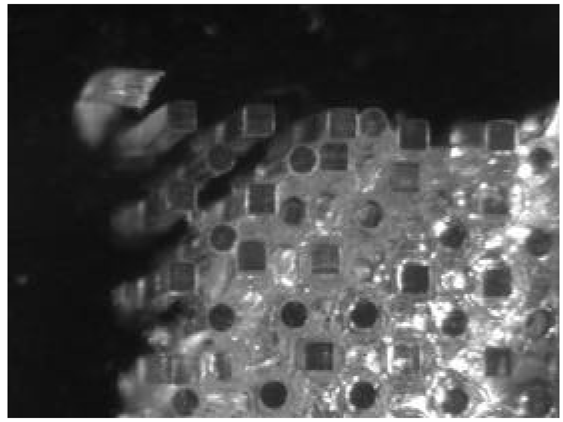
a) Diffusion from large block of SU-8 into field of posts

b) Diffusion of acid near crosses

Figure 36: 4.5 mm sample with bottom dose of 20 J/cm^3.



a) Large Diffusion from SU-8 block into field of posts



b) Diffusion covering all but one corner of posts

Figure 37: 4.5 mm with a bottom dose of 30 J/cm^3.

From these results it is conclusive that the new procedures to limit diffusion do dramatically improve results. As exposures become more and more challenging it will be necessary to further investigate diffusion of PAG in SU-8 and to further optimize the SU-8 processing steps. The first thing that should be done is to create a model that accurately predicts the diffusivity of PAG SU-8 during all of the SU-8 processing steps, taking all variables into consideration. Once this is done it will be possible to use the created model to further optimize the SU-8 manufacturing procedure. .

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Vita

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